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THE INSTITUTE OF PETROLEUM

AN Ordinary General Meeting of the Institute of Petroleum was held at Manson House, 26 Portland Place, London, W.1, on January 9, 1952, the President, Mr C. A. P. Southwell, M.C., in the Chair.

THE PRESIDENT, opening the meeting, said: It is my pleasure to introduce to you Mr E. C. Masterson. He is in charge, in London, of the field operations of the Kuwait Oil Company, having previously had a wealth of experience in America, Roumania, and other places.

I should like to take this opportunity of saying that the loss of the Roumanian oil industry has been the great gain of the Kuwait Oil Company in its rather spectacular development. At the time we were building up the Kuwait staff we were very fortunate in being able to obtain the services of experienced men from Roumania, of whom Mr Masterson is one.

I should like to express a welcome on behalf of the Institute to Mr C. A. Swigert, president of the Trans-Arabian Pipe Line Co., who is in London on a short visit. I hope Mr Swigert will be able to give a talk to the Institute when next he is in London, as we should welcome very much a talk on the engineering problems connected with the Tapline project.

Mr E. C. MASTERSON then presented the paper which follows, first explaining that it had been prepared jointly by Mr Boaden and himself. The task of presenting it had fallen upon him, as Mr Boaden had returned to Kuwait after summer leave in Britain.

SOME ASPECTS OF FIELD OPERATIONS IN KUWAIT

By E. BOADEN, O.B.E.* (Fellow), and E. C. MASTERSON * (Fellow)

IT is proposed to first deal very briefly with the early stages of field operations in Kuwait and to assume a good general knowledge of the geographical position of the Arab State of Kuwait, and of the conditions which prevail there.

This paper deals only with field operations, and does not include discussion of problems affecting the shipping of oil, nor indeed any of the numerous and allied operations such as housing, communications, industrial installations, etc., which have necessarily occupied great attention, and which have been complicated by the fact that practically all needs of the operating company have had to be imported. When considering the extent and scale of operations in the oilfield, it is as well to remember, first, that these involve intense activity in other directions, particularly in the provision of community requirements for a large number of employees of different nationalities and modes of life and, secondly, that the supply position has never been free from difficulty.

The period of development to be discussed is from 1945 to the present

* Kuwait Oil Company Ltd.

day. Although nine wells were drilled on the Burgan structure between 1938 and 1942, when operations were suspended on account of the war, no

TABLE I

Production in the Burgan Oilfield

	Tons
1946	797,350
1947	2,185,309
1948	6,291,577
1949	12,183,669
1950	17,018,666
1951	27,783,170

crude oil was exported from Kuwait until June 1946. From 1945 to the present date 103 additional wells have been completed.

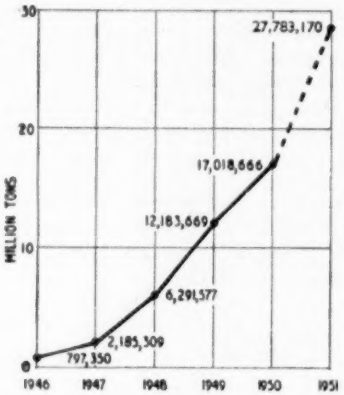


FIG 1

PRODUCTION IN THE BURGAN FIELD

The rate of development of the Burgan field can best be illustrated by the production figures in Table I, which are plotted in Fig 1.

The original development of the field was to a pattern of one well for each 600 acres, the distance between wells being just over 1 mile. This development enabled the limits of the field to be established by the beginning of 1950. On its completion "infill" drilling was started on the basis of 200 acres for each well.

The field has been divided into areas capable of producing between 100,000 and 120,000 b.d. In each of these areas, a "gathering centre," or gas-separator installation, has been constructed capable of processing this amount of oil. From these collecting points, the oil is pumped into a pipeline transit system through which it is conveyed a distance of 9½ miles to the Company's main storage tanks, located at Ahmadi on a ridge some 430 ft above sea-level. From this point, the oil flows by gravity through five 22- to 24-inch diameter pipelines to the oil-loading pier at Mina al Ahmadi.

Geology

The Burgan oilfield covers an area of approximately 135 sq. miles. The structure is an elongated dome, with a major north-to-south axis 12 to 15 miles in length, and a minor east-to-west axis 8 to 10 miles in length. The oil is found in rocks of Middle or Lower Cretaceous age. In contrast to Persian and Saudi Arabian fields, in which the normal reservoir rock is limestone, the producing formations of the Burgan field are of sand and sandstone. The depths to the producing horizons are relatively small with no well exceeding 5000 ft. The producing section is made up of four major sand bodies, separated from one another by shaly intervals, and the oilfield as a whole is subdivided into two major reservoirs by an intervening limestone interval, generally oil-stained, but not productive. The entire producing series is underlain by salt bottom water; the oil/water contact is practically horizontal and cuts across each individual producing sand. The sand thicknesses differ slightly from well to well, and the amount of unproductive lime and shales vary between rather wide limits.

From the foregoing, it will be clear that as the flanks of the structure are reached, the fourth sand is no longer productive, whereas good yields can still be obtained from the sands above. Still further outwards the third sand approaches the water table and becomes waterlogged, and production possibilities are confined to the first and second sands above the intervening limestone. The third and fourth sand pays are composed of fine- to medium-grained sand and sandstone, unconsolidated and interbedded with laminations of siltstone and shale, whilst the first and second sand pays consist of fine-grain, rather calcareous and well-cemented sandstone with shaly laminations.

Structure

To the petroleum engineer, Burgan is in many ways an ideal structure, since most of the faulting present in the field is of a minor nature, and only one fault interferes in any way with production characteristics. It has the very great advantage that, although a sand field, its wells will produce at high rates without making sand. In addition, the pressures in the producing formation are not excessively high and do not cause undue difficulties during drilling operations. All the usual precautions, such as mud control, and the use of blow-out preventers are, of course, taken, but the wells can be kept under control without difficulty.

Field Development

Practically the whole of the drilling programme in the Burgan field has been carried out using National Type 50 rigs. As many as twelve of these were employed in 1949 when drilling activity reached its peak. Once the well potential became sufficiently large to satisfy the capacity of the existing gathering centres, two National 50 rigs working at greatly improved efficiencies were able to complete wells at a rate sufficient to keep pace with additions to separator and other oil-handling facilities.

Skidding of Rigs

The nature of the desert terrain at Kuwait makes it possible to skid the derrick and draw-works, as well as the engine and their sub-structures

with other components, from one site to another by using up to ten or even twelve D8 caterpillar tractors. This has resulted in a great saving of time, especially during and since the latter half of 1950, when the rigs in use have been " unitized " by mounting the two G.M. diesel engines on a cantilever extension built on to the 136 ft × 24 ft Parkersburg derrick at a draw-works level of 14 ft 6 inches. Two vertical 6-inch supports are hinged to the outer end of the cantilever and fitted with screw jacks, which serve to steady and hold some of the weight of the engines during drilling

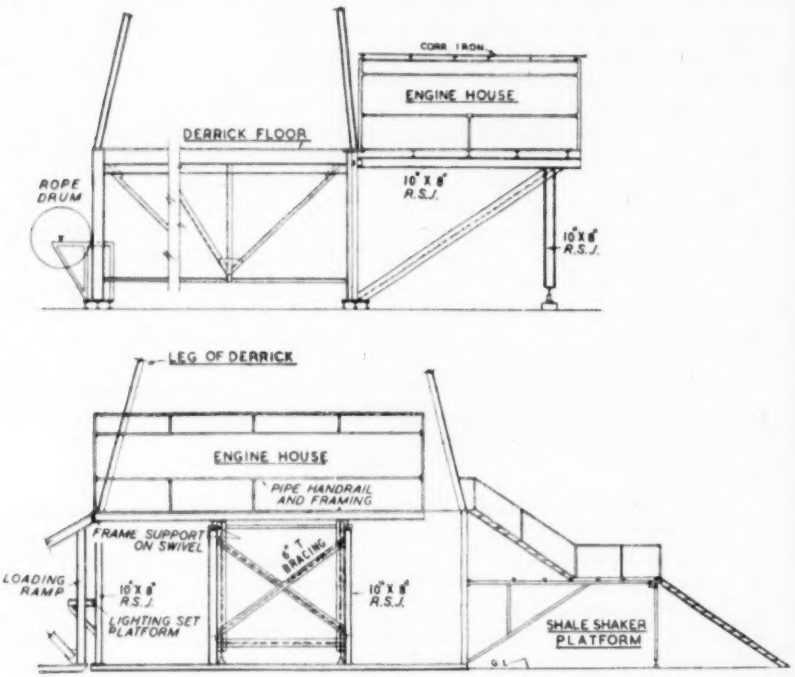


FIG 2

UNITIZED RIG

Elevations on east (*above*) and north (*below*) sides

operations. The shaker screen and spare drum line are also permanently mounted on cantilever brackets and move with the derrick. As soon as a well is completed and before the well has started making clean oil, the rotary table is lifted forward, the front end of the draw-works-engine unit lifted a few inches with the aid of the block, and the whole unit pulled forward off the cantilevers into the derrick base using the cat-head of a winch truck. The jacks at the lower ends of the cantilever supports are slackened off and the supports folded into the derrick. Ladders, which are hinged to the derrick, are folded up, a sub-structure tie bar is removed to allow room for the flowhead to pass, and the derrick is ready for skidding. Details of unitization are shown in Fig 2.

All rigs are provided with two C-250 National slush pumps, each mounted as a skid unit together with two twin G.M. 6/71 diesel engines. These are easily transported on winch trucks. Manifolding is "unitized," and pipework is flexible and fitted with quick union connexions. Pipe-racks and mud tanks are skid mounted.

Unitization of the rig has reduced the number of separate unit moves to ten, and this is the order in which the various units are moved :—

1. The mud settling tank.
2. Pump manifold platform together with suction tank and mud storage tank (skid).
3. Mud pump No. 1.
4. Mud pump No. 2.
5. Mud mixing platform.
6. Mud chemicals platform.
7. Drilling water tank.
8. Engine fuel tank.
9. Derrick, engine house, etc. (skid).
10. Loading ramp.

The above order of moving enables the pumps, tanks, etc., to be assembled at the new site while the derrick is being skidded.

The skidding arrangements as outlined have resulted in a reduction of the time taken in rigging down, moving some 2 or 3 miles, and rigging up to less than 24 hr per well.

It is true that systems have been developed entailing the raising of the derrick on to track waggons whereby such moves can be carried out with the use of only two tractors. However, since the tractors were available, it was found that moves could be completed in a much shorter time by using the method described than if a system requiring special conveyors was adopted. With this latter type of equipment, heavy-lift hydraulic jacks have to be used to raise the derrick on to the waggons. Observation of a contractor working on such a move during a recent visit to Venezuela showed that it took approximately 3 hr to raise the derrick on to the track waggons in readiness for the move. If the country over which the move has to be made is rougher than is generally encountered in Kuwait, the use of special tracks presents a distinct advantage.

Casing Programme

The normal casing programme which has been adopted in Burgan is as follows : 16-inch 75-lb welded casing is set in 20-inch surface hole at about 300 ft, $10\frac{3}{4}$-inch 45-lb casing in $13\frac{3}{4}$-inch hole at about 3250 ft and a 7-inch 23-lb oilstring through the pay zones at about 4750 ft. All strings are cemented to the surface. The 7-inch string is fitted with centralizers and scratchers, and is reciprocated whilst being cemented. Electric logs and caliper surveys are made before running the $10\frac{3}{4}$- and 7-inch casing strings, and temperature surveys are used to assess the value of the cement jobs. Where lost circulation zones prevent cement returns to the surface, extra cement is batched down the annular space until the temperature surveys show that the job is satisfactory. The $10\frac{3}{4}$-inch casing seat is pressure tested on drilling out.

Overcoming Circulation Losses

Drilling operations have presented no particular problems, excepting difficulties connected with lost circulation zones. There is generally no special difficulty in penetrating the sands and gravels down to the 16-inch casing seat. The hole is spudded in, using a medium-weight mud, salvaged from previous wells, possessing the good plastering qualities required to hold up the soft sands found near the surface. Mud is occasionally lost in this upper hole, but this presents no real problem.

Below 300 ft the intermediary hole can, however, lose mud in any quantity at any point down to the casing seat at about 3250 ft. No correlation of lost circulation zones from well to well has been possible, but the major zones appear to lie between 600 and 800 ft in the Eocene limestone and between 3000 and 3200 ft in the Upper Cretaceous. The lower part of this latter limestone is dense in texture, and the policy is to set the intermediary string in it, with a reasonable certainty that the major trouble will be safely cemented off behind it.

Considerable sections of the Eocene and Cretaceous limestones are of a cavernous nature and are waterlogged, necessitating a fine balance being maintained between the mud weight and the formation pressure, in order to prevent total loss of circulation on the one hand, and strong water flows on the other.

Numerous experiments have been tried, including the use of oil emulsions, to find a set of conditions which would allow equilibrium to be maintained, but none has proved more than a palliative. Many types of sealing materials have been tried and, on occasions, some have given valuable aid, but in all difficult cases the use of cement plugs is necessary. This can be a lengthy and expensive process, as was shown in the case of a well drilled in the spring of 1950, which required a total of fifty-one cement plugs at various levels before casing depth was reached. Altogether, nearly 9000 sacks of cement and 1000 sacks of sealing materials were used at this well, in addition to several tons of rope, burlap, matting, broken bricks, and sewage pipe.

During the last few months, the technique employed in drilling the intermediary hole has been changed with good results. The use of mud in this section has been abandoned, and instead, the maximum quantity of water up to the full capacity of the well pumps is used when zones of total or partial loss of returns are met. Drilling is continued, and it is rarely that the cuttings do not seal off the porous zones comparatively quickly. If they do not, there is no alternative but to place a cement plug.

Recently thirteen plugs were placed in a well totalling 1600 sacks of cement without obtaining full returns. At this stage it was decided to try gravel. This was mixed with a fairly heavy consistency of bentonite and dropped into the drill pipe by hand. This mixture was displaced with mud and immediately followed up with fifty sacks of cement. After setting for 12 hr normal drilling was resumed with full returns.

A flow of sulphurous water is sometimes encountered, and this normally helps the situation, but care has to be taken that the hydrogen sulphide, which is present in this flow, is effectively dispersed.

After setting the 10¾-inch intermediary string of casing, the use of water

is continued to within 150 feet of the first sand, from which point drilling continues, using an oil emulsion, phosphate-base mud weighing about 11·2 lb/U.S. gallon and with a water loss not greater than 5 cc.

The practices described have contributed greatly to the speeding up of drilling operations and, together with the elimination of the use of mud over a long section of the hole, have resulted in lower costs. For example, in early 1948, the cost of mud and chemicals for each well was of the order of $20,000, in 1949 $12,000, in 1950 $6000, and is not expected to exceed $3000 in 1951.

Completion Times

As a result of the measures discussed, there has been a very marked improvement in completion times of Burgan development wells, as is shown in Table II.

Completion Practices

It is the practice to run electric logs and caliper surveys before setting the intermediate and oil strings. Casing is normally cemented to the surface. To ensure a good cementing job, the oil string is fitted with centralizers and scratchers and is reciprocated during cementing. Temperature surveys are then made to assess the efficiency of the cementing job. Where lost-circulation zones prevent cement returns to the surface, additional cement is batched down the annulus until the temperature survey shows that the job is satisfactory.

Wells are completed as dual producers, with production from the lower zone obtained through tubing, and from the upper zone through the casing in which a suitable packer has been set to isolate the two sands. Wells in the centre of the field are completed in the third and fourth sands, and wells on the flanks in the first and third.

To prevent water coming in and to allow a reasonable water-free flowing life of the well, an interval of at least 100 ft is left unperforated above the oil/water level, excepting where a shale break intervenes, in which case this interval is reduced to 75 ft. A close study of the electric log determines the intervals to be perforated, and where shale breaks occur within the producing sand, 10-ft or longer intervals remain unperforated for future packer setting points and water control.

Until recently, it has been the practice to perforate the oil string with a Schlumberger 100-mm gun using 8·5-mm bullets at six shots per foot. This density of perforation required between 2000 and 2400 bullets per well. To save time, two guns are made up in assemblies 24 ft in length and run and reloaded alternately. On completion of the perforating operation, a rock bit with cut-away water-courses is run, the perforations scraped, and circulation reversed to recover any bullets which may have failed to penetrate the casing.

There have been failures of up to 5 per cent and, in one or two particularly bad cases, nearly 10 per cent. Where the number of failures is less than 5 per cent, no further action is taken. If it is more, however, it is the practice to reperforate with a density of shots depending on the number of failures.

Shaped charges have been used recently, and it would appear that as

TABLE II

Completion Times—Burgan Wells

Well no.	Date completed	Feet drilled		Time, days							Rate, feet per day	
		Surface to intermediate depth	Intermediate to total depth	Total	Rig up rig down move	Spud to resume drilling after casing and cementing intermediate pipe	From intermediate depth to T.D. laying down D.P. period casing and cementing drill plug	From drilling plug to release of rig	Drilling from spud to intermediate depth only	Drilling from intermediate to total depth only	From spud to intermediate depth only	Intermediate to total depth only
BG27	Aug. 1948	3350	1341	90·7	10	33·7	29·0	18·0	27·0	23·0	124·0	59·0
BG55	May 1949	3570	1187	65·9	8	35·6	13·3	9·0	31·0	12·0	115·0	99·0
BG76	Oct. 1949	3407	1255	70·6	8	32·3	26·3	4·0	26·0	24·0	131·0	53·0
BG95	Feb. 1950	3395	1316	84·6	12	43·0	14·6	15·0	38·0	18·0	89·0	73·0
BG99	Oct. 1950	3290	1433	53·1	4·9	31·4	12·8	4·0	27·1	8·3	121·2	173·5
BG101	Jan. 1951	3318	1445	40·2	3·2	19·8	14·2	3·0	15·3	10·7	217·0	135·0
BG103	Feb. 1951	3261	1486	39·1	1·1	18·6	16·2	3·2	14·8	10·2	220·5	145·6
BG106	April 1951	3280	1473	27·9	1·3	13·5	11·1	2·0	12·2	6·8	268·9	216·6
BG109	April 1951	3294	1436	24·8	1·5	12·4	9·4	1·5	9·7	5·3	339·5	270·9

good, and more consistent, results are obtained with four shaped charges per foot compared with bullet perforations at a density of six per foot. The use of shaped charges has resulted in a saving in perforating costs of about 10 per cent.

After perforating and scraping, these dual producing wells are completed by running a Lane Wells circulating packer on 3½-inch O.D. tubing with a Gray type 4000 p.s.i.g. well-head Christmas-tree with Cameron wings on the casing zone. Wells are brought into production by washing, and the packer is set and tested in the unperforated interval between the two selected zones.

Coring

As stated earlier, the Burgan field was originally drilled on a 600-acre hexagonal grid. One well out of three on pre-determined sites was cored through the producing formation, and considerable core information is thus available for use in the correlation of the 200-acre spacing infill well now being drilled. Coring was done by conventional methods and, in these relatively unconsolidated sands, overall recovery was about 50 per cent.

In recent completions diamond coring with oil-emulsion mud was introduced with very satisfactory results. This has now become our standard practice. Recovery has averaged about 90 per cent, and on occasions has been as high as 100 per cent. In a recent well twenty-six cores were taken over an interval of 896 ft with a total recovery of 87·7 per cent.

In the core barrel selected the core catcher is attached to the inner barrel, and so does not rotate as the core is being cut. Therefore, the catcher and the core are less likely to be damaged, as is the case where the catcher is housed in the outer barrel.

Apart from the improved recovery, the cost of diamond coring is lower than conventional coring because of the much greater footage obtained from the diamond cutter heads. In one well as much as 904 ft was cored with one head, and the head was still fit to run again. Comparative costs based on the difference in the cost of the cutter heads, all other factors being equal, show that diamond coring is about 100 per cent cheaper than conventional coring. In one case the cost of the diamond heads worked out at 13*s* 4*d* per foot as compared with 27*s* 4*d* per foot for the conventional heads.

A very complete core laboratory has been provided to permit complete examination of the cores. Analysis of vertical and horizontal permeability, porosity, and oil and water saturation is made of each foot of core recovered.

Deep Test Well

In addition to the 112 normal producers at present completed in the Burgan field, mention must be made of the deep test well which, after reaching a total depth of 13,853 ft without favourable result, was finally abandoned as an exploratory well and converted into a normal Burgan producer. This well was drilled near the top of the structure, using a National 125 type rig with 136-ft derrick, powered by three Superior

375-hp engines also rear driving an Oilwell 220-P type slush pump. A second 220-P type pump was powered by two further Superior 375-hp engines, and a sixth engine of the same type was used to drive an independent National C-250 pump for mud-mixing purposes. A Brantly feed control was a feature of the rig. 20-inch surface casing was set at 299 ft, 13⅜-inch at 3557 ft, 9⅝-inch extreme line at 4954 ft and 7-inch extreme line casing at 8942 ft. A 5⅞-inch hole was carried to total depth. Considerable diamond coring was done at depth with excellent recoveries of up to 100 per cent. A number of drill-stem tests were carried out below the 7-inch shoe. It is of interest to note that certain very tight formations at 10,153 ft, containing minute quantities of gas and oil registered a bottom-hole pressure of 6375 p.s.i.g. For this reason, mud weighing up to 17 lb/U.S. gallon was used in drilling below that depth.

Production Practices

Flowlines

Although a number of early wells were provided with 4-inch flowlines leading to gathering centres and some of these were doubled at a later period, the standard flowline provided at each well is now of 6⅝-inch O.D. seamless linepipe tested to 2000 p.s.i.g. The lines are laid on the surface and buried only at intersections, road crossings, and the like. The capacity of an average flowline of 9000 ft in length is about 9500 b.d. of stock tank crude. This is on the basis of a well-head pressure of 550 p.s.i.g. and a pressure drop of 100 p.s.i. along the line. A group of fourteen to twenty wells flow into each centre. Their individual flowlines are provided with adjustable chokes followed by orifice meters, from which all crude passes into a three-way manifold at the gathering centres. Operators are thus enabled to control any well from the gathering centre itself.

Gathering Centres

At the present time the Burgan field has six gathering centres in operation and a seventh under construction. Once the potential of the Burgan wells became known, consideration had to be given to the size of the separating stations or gathering centres as they are more commonly called. The decision was influenced, not only by the potential of the wells, but also by the maximum length of flowlines which could be used for any individual well. It was found, for instance, that production from wells could be handled with 6-inch flowlines up to about 3 miles in length, but when the length increased beyond that friction losses caused loss in production. At the same time it was felt that the installation should not be too large, on account of the hazards involved. After due consideration, a capacity of 100,000 b.d. was chosen as ideal for Kuwait conditions.

When the capacity of the gathering centres was agreed, it became necessary to decide upon the type of separator to be installed and the number of stages of separation. In considering the type of separator to be adopted there seemed to be considerable advantage in favour of the horizontal rather than the vertical type more generally in use in the States. In a vertical separator, the surface of the liquid from which dispersion of gas takes place is limited to its cross-sectional area. Flow and counter-flow of rising gas and falling

liquid is liable to carry over a certain amount of liquid in the gas stream. A slightly tilted horizontal separator seemed to be the answer to our problem. In such a vessel the crude oil is admitted in the centre, the gas outlet is at the raised extremity, and the separated crude outlet at the lower end. The admission of crude oil in the centre of the vessel leaves the upper portion free for the full passage of gas. The particles of liquid entrained in the raised portion of the vessel when falling are not in conflict with the gas released from the liquid in the lower part of the vessel. The amount of tilting appears to have some influence on the capacity of the installation, and we adopted a policy of installing these vessels at an inclination of 1 in 30.

In considering the number of stages of separation, tests showed that little is gained in the quality of the crude by increasing the number of stages from five to seven, but since there *is* a slight improvement and the amount of steel in the installation is not affected (this being a function of its capacity) seven stages were eventually selected. It is interesting to note that it has been proved that a seven-stage unit will yield approximately 1·6 per cent additional liquid compared with three stages of separation. On a 100,000-brl installation, this is equal to 1600 b.d. The improvement in the gravity of the crude as between three and seven stages is approximately 0·5° API or 0·0026° in specific gravity. Such an improvement can put the production into a higher-gravity group, which, at 2 cents per barrel per degree API, can result in a considerable increase in income.

Tests have shown that, if the gas is separated from the oil by flashing directly to atmospheric pressure, only 39 per cent of the total *iso*pentane (*i*-C_5) is recovered. In a three-stage unit, the recovery is increased to 75·5 per cent, while in a seven-stage unit this increases to 83·5 per cent.

Now for a brief description of the installation (Fig 3) :—

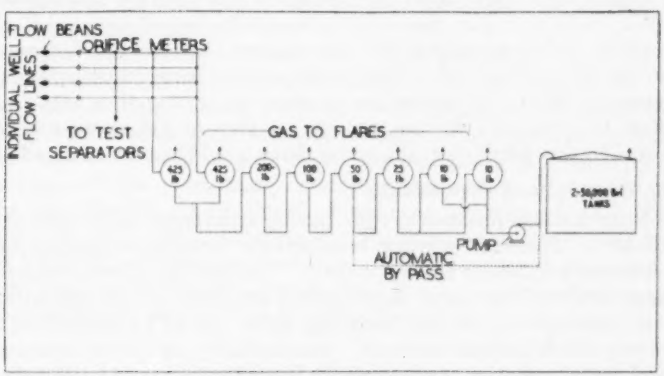


FIG 3

SEPARATOR FLOW DIAGRAM

There are eight separators to each gathering centre. These individual vessels are larger cylinders, 145 ft long and 4 ft 6 inches in diameter. The first stage is operated at 425 lb, being governed by the flowing pressure of the wells. There are two high-pressure separators working in parallel

at this stage. Here about 60 per cent of the total gas is taken off with a gas/oil ratio of 500 cu. ft/brl; this represents about 30 mill. cu. ft. of gas per day. The following five stages of separation operate at 200, 100, 40, 25, and 10 lb respectively. The crude oil receiving tank acts as the seventh, and final, stage. Two separators are provided at the 10-lb stage to handle the large gas evolution at this low pressure. From this stage the crude is pumped by centrifugal pumps into storage tanks at substantially atmospheric pressure. These transfer pumps are driven by gas turbines which receive high-pressure gas from the first stage of separation, after it has passed through pre-heaters. A by-pass is provided from the 50-lb separator so that oil can be transferred to storage from this stage in the event of a breakdown of transfer pumps.

It is not possible to obtain individual well characteristics from the main separator installation, because all wells are joined into a common manifold on the upstream side of the installation. To provide for this, however, three small test separators are installed at each centre. It is possible, at any time, to run any individual well through this test installation and obtain all flowing characteristics.

As far as tankage is concerned, each centre is provided with two 50,000-brl tanks for normal operation, and two 10,000-brl tanks for testing individual wells through the pilot installation already described. Production is allocated to wells on the basis of meter readings, these figures being checked from time to time by flowing any particular well for a period of a few hours through the test installation.

Well Potentials

Although individual well production-capacities vary, the average first and third sand well may be expected to produce some 5000 brl daily, whilst a third and fourth sand dual producer will produce about 9000 b.d. Individual wells and pay zones are periodically tested for gas/oil ratios. Productivity index measurements are routine, as also are bottom-hole-pressure surveys. There is a considerable margin between present bottom-hole pressures and crude saturation pressure which, together with the rate of bottom-hole-pressure decline, promises a long period ahead free of gas troubles. In this particular, the water drive would appear to be efficient.

Gravity, Porosity, and Permeability

The Burgan sands produce a crude having an average stock tank gravity of 31·8° API. There is, however, considerable variation in gravity according to depth of production below sea-level. The gravity of crude drawn from the three sands at the same depth below sea-level is approximately the same and, through the normal producing series, the API gravity falls about 1° for every 200 ft increase in depth. Permeability, as can be expected in a sand and shale formation, varies greatly, and is as low as 384 millidarcies in parts of the upper sands, and ten or even fifteen times as great in some of the lower sands.

Pump-stations and Pipelines

At each of the gathering centres a transit pump-station has been constructed in which three gas-engine-driven pumps handle the normal production of the centre.

The pumping units are driven by 8-cyl Cooper–Bessemer gas engines through a speed increaser. Natural gas is used as fuel. The engines develop 675 b.h.p. at 450 r.p.m. Each pumping unit set consists of a boosting pump delivering through two lubricating-oil coolers to the suction of the main pumps at 50 p.s.i.

The main pumps have a capacity of 730 Imp. gal/min at 60° F against a discharge pressure of 800 p.s.i.g. These operate at 3250 r.p.m. being stepped up to this speed by a Farrel Birmingham speed increaser from 450 r.p.m., the speed of the engines.

Jacket water cooling is provided by Alco radiator-type cooling towers. It is a closed system with a view to reducing water consumption to a minimum.

Two 125-kW generating sets driven by Cooper–Bessemer dual-fuel engines provide the necessary power for driving all auxiliaries including air compressors for starting, water-circulating, and lubricating-oil pumps.

To provide flexibility in the movement of crude from low-pressure and test separators, four transfer pumps are installed in each pumping station. These are driven by gas-expansion turbines, the gas, which is preheated before use, being taken off at the right pressure from the appropriate separator.

In a more recent completion gas-expansion turbine pumps are being installed as main pumps in place of the standard engine units referred to above.

The turbines are single stage, each developing 950 b.h.p. at 6000 r.p.m. They are driven by natural gas at 250 p.s.i.g. and a temperature of approximately 250° F. The quantity of gas required for each turbine is about 13 million cu. ft/day.

The gas will be taken off from the first-stage separators, passed through mist extractors and scrubbers, and then through preheaters to bring it up to the required temperature.

The pumps are nine-stage " Plurovane " type, geared down to 1520 r.p.m., with a capacity of 1430 Imp. gal/min against a head of 810 p.s.i.

From each of the gathering centres, the oil is pumped through 12¾-inch collecting lines into the main pipeline system running northwards from the centre of the field. Two 20-inch lines convey in excess of 700,000 brl daily, maintaining, at the same time, working pressures at all transit pump-stations below the 750-p.s.i.g. limit. From the northern end of the field the two main 20-inch transit lines convey the production to the tank farm located some 9½ miles to the north-east. The capacity of these two lines has now been nearly reached, and a third line is contemplated to permit the handling of the higher rates of production which are envisaged.

Storage

The crude-oil storage tanks in the main tank-farm area consist of twenty-one tanks varying in size between 139,000 and 168,000 brl. They are all of the floating-roof type. For more efficient operation, motorized valves are being installed on all tanks, the operation of which will be controlled from a specially constructed control building. In addition, following upon the installation of liquid-level gauges and automatic thermometer recorders,

tank levels and oil temperatures will be continuously recorded on instruments mounted in the control room. These facilities will enable the more rapid switching of tanks, with a consequent increase in tanker-loading efficiency. A careful study has been made of storage capacity required, and there is probably no other field where such high production rates are handled with so little storage. In this connexion, it must be remembered that Kuwait has no pipeline outlet, as is the case in Saudi Arabia (Tapline);

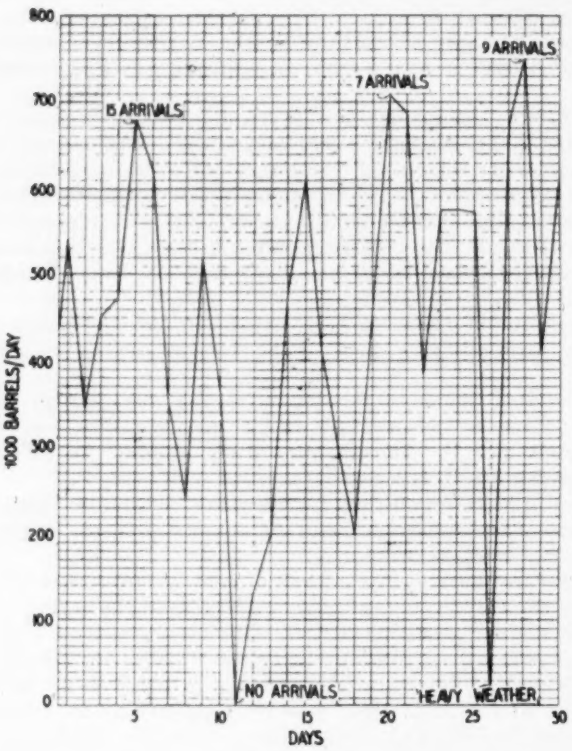


FIG 4

VARIATION IN DAILY SHIPMENTS

the entire production is exported by tanker. This means that, even with the most careful planning, the daily outlet is irregular due to non-arrival or bunching of tankers. It is necessary, therefore, to store the crude when tankers do not arrive or cannot come alongside the berths due to bad weather. Other factors influencing the amount of storage which must be provided are: (*a*) the size of tankers, and (*b*) the pumping rate from the field. It is fortunate that in Kuwait production is free from impurities and no settling time is required. If this were not so, additional tankage would have to be installed.

Fig 4 shows how shipments can vary from day to day for the reasons

already given. Daily loadings during the particular month shown reached a maximum of nearly 800,000 brl on one day, whilst being at a complete standstill on another.

With such limited storage it has been of primary importance to operate the tank farm efficiently so that, when tankers arrive, loading operations can be carried out as speedily as possible. With this in view, electrically operated valves are being installed on all tanks. Automatic liquid-level gauges and thermometer recorders are also being added. In this way the operation of the valves, the reading of tank gauges, and the taking of oil temperatures is made from instruments mounted in a tank-farm control building.

It is not wished to imply that sufficient storage is available or that it has not been necessary to pinch back production on occasions for want of storage. But, under the very heavy construction programme, it has been necessary to economize effort in some directions, and it was felt that this could best be done in the matter of storage by utilizing available capacity at maximum efficiency.

Mention should be made of the Mina al Ahmadi jetty, of which the company is very proud. The main structure of the new port is a loading pier which is believed to be the largest of its kind yet built. Of steel construction and stretching 4100 ft out to the open sea, it carries a roadway 24 ft wide designed to take the heaviest motor transport and a pipeway comprising eight crude-oil loading lines of 24-inch diameter, together with a number of lines of smaller capacity for fuel oils and water for ships and compressed air for cranes and hose derricks. The northern extension of the pier head, 2800 ft long and 105 ft wide, has six oil berths at which tankers can be loaded at a high rate, while the southern extension 1050 ft long and 100 ft wide, provides two berths for cargo vessels. Some 20,000 tons of steel and between 400 and 500 miles of timber for decking have been used in the erection of this pier, which was completed in eighteen months from the time that the first pile was driven into the sea-bed.

General

In the foregoing an attempt has been made to describe briefly some of the most important aspects of the development of the Burgan oilfield.

It has not been possible to include a discussion of the many other problems affecting the shipping of oil, which have necessarily occupied great attention, and which have been complicated by the fact that practically all the requirements have had to be imported. When considering the extent and scale of operations, it is as well to remember that these involve intense activity in other directions, particularly in the provision of community requirements for a large number of employees of different nationalities and modes of life, working under somewhat trying conditions, particularly during the summer months.

Throughout the post-war development period the supply position has been one of the main concerns. Just as the situation was starting to ease, as a result of industry returning to normal after six years of war, the new rearmament programme both in the U.K. and in the United States has once again made the supply position difficult. The rate of development in Kuwait must, therefore, be appreciated in the light of these considerations.

Discussion

The President : I am sure that we all appreciate very much the paper and would like to thank Mr Masterson for his interesting presentation of it. He has covered a very wide field in a short time ; therefore, I hope there will be a good discussion and that more details of these operations will be brought to light as a result.

We have with us to-night Mr A. C. Hartley, the president of the Institution of Mechanical Engineers and of the Anglo-Iranian Oil Company. We in Kuwait will never forget that we had the Gulf Company and the Anglo-Iranian Oil Company behind us with a team of technical men whenever we required them.

We are also glad to have with us Mr Heap, the representative of Bechtels, who built the oil pier which Mr Masterson has briefly mentioned. I am sure his firm are as proud of its success as we are.

A. C. Hartley, C.B.E. : We have had described to us what I think may well be claimed one of the most remarkable developments in the oil industry or, indeed, in any other industry in the world to-day. I think that Mr Masterson and Mr Southwell and others have been so close to it that they cannot fully appreciate what has been done.

I had the privilege of going to Kuwait for the first time in February 1946 ; oil was not produced there until June 1946. It was then obvious that there would be very great development there, due to the collaboration between the British and the Americans concerned, the Anglo-Iranian and the Gulf Oil companies. One was impressed by the remarkable team-work, everyone being determined to produce oil as soon as possible.

I had the pleasure of again visiting Kuwait in October 1947. Production at that time was very nearly at the rate of 100,000 b.d. But the borings warranted a very great extension, and subsequent events proved how right was the view taken of the prospects. A programme had been prepared in May 1947 calling for 250,000 b.d. in 1949, nearly 400,000 in 1950, and 488,000 in 1951. But in August of that year it was felt that that output could not be achieved, so the figures were lowered to 195,000, 295,000, and 360,000 b.d. in those years. But the first series of figures has been attained. In 1949 they nearly achieved the target, the production being 240,000 b.d. ; a few weeks ago the average was 560,000 b.d., and it is increasing all the time.

The last visit to Kuwait was made at Mr Southwell's request to see how the partners could help in the drilling and construction, in the development of schemes, and in the provision of staff and materials. It was a very great privilege to have been associated with the work at that time, and I appreciate hearing from Mr Masterson what an absolutely magnificent achievement it has been.

A matter which has impressed me is the determination of the Anglo-American team to overcome every difficulty in the most direct way. For instance, where there is mud loss they push the drill ahead and pump and pump to the full capacity allowed by the available water supply and often succeed in sealing up the porous zones with the drilling cuttings, thereby effecting a tremendous saving as compared with conventional methods.

Again, in America vertical separators were generally used. In Persia the separators were horizontal, but now they are at 30°—a very good compromise. In some cases they have put the vertical separator at the end of the horizontal one.

CHARLES DABELL: May I ask what happens to the 50 million cu. ft. of gas?

MR MASTERSON: Apart from the quantities required for our own industrial and domestic requirements, the rest of the gas is flared. We are, however, examining the situation very actively with a view to using the gas either for repressuring, gasoline recovery, carbon-black manufacture, or a combination of these. We hope that before too long we shall be able to put out the flares, or at least, reduce their size appreciably.

R. L. MESSENT: Is the present production balanced by a combination of the water influx and gas-cap expansion? If not, what type of secondary recovery is contemplated?

MR MASTERSON: We feel that we have a fairly effective water drive. However, we are watching the behaviour of the reservoir very carefully, and more especially during the last few months, during which production has been at the present high level. A study of conditions as they develop may indicate the advisability of water or gas injection, or a combination of both, at some future date.

R. H. W. HAMILTON: I personally am very interested in what Mr Masterson has said about the movement of rigs, particularly by skidding, for we in the Iraq Petroleum Co., are studying that matter. I think our rigs are rather heavier than those to which Mr Masterson has referred, but the terrain is much the same.

During the past few years we have had considerable trouble with lost circulation, and I certainly agree with Mr Masterson that, if the trouble becomes really bad, the only thing to do is to use cement in a big way. But in certain cases where large quantities of water have been available (particularly when we have been able to put pump suctions into the sea itself) we have pumped in large volumes of water—volumes of perhaps 25,000 to 30,000 b.d.—and have been able to carry on drilling in that way rather than by putting in cement. I would like to ask whether, in the drilling in Kuwait, the technique of using very large quantities of water has been applied at any time.

MR MASTERSON: In regard to the circulation losses, I mentioned in my paper that we can generally overcome these by drilling ahead with pumps wide open, pumping in as much water as the pumps will handle. I have no figures regarding the rates of water input: as long as the bit can be kept free and the table rotating we keep on drilling. If this is not possible, we must resort to one or more cement plugs.

During my recent visit to the U.S. I found that they were having considerable success in overcoming circulation losses by the use of the diesel oil/bentonite squeeze method. This involves suspending a very high concentration of bentonite in diesel oil (about 300 lb of bentonite

per barrel of diesel oil); a small amount of sealing material may also be included. The suspension is squeezed into the formation, together with mud in the hole, which is displaced down the annulus. The squeeze ratio is roughly 1 brl of mud to 4 brl of diesel oil and bentonite. The resulting mixture becomes very viscous, as the bentonite extracts the water from the mud and increases in viscosity as it is displaced into the formation under pressure and comes into contact with more mud. The diesel oil is squeezed out under pressure, leaving the solid, partially hydrated bentonite behind. Any additional filtrate or mud entering the mixture will cause further swelling and hardening.

We propose trying this method in future troublesome cases.

H. C. H. THOMAS: You have mentioned that with four shaped charges per foot, you are getting much the same results as with six bullet perforations per foot. Can you say why that is so, and how the comparison was made between the results of using four shaped charges or six bullets per foot or, in fact, any other number of either type of perforation?

MR MASTERSON: The reason is that you get deeper penetration and no failures.

We have carried out tests and comparisons between cored wells which have shown similarity in sand thicknesses, compositions, etc., and our findings are based on the results of these tests.

R. L. MESSENT: You refer to the less consolidated 1st sand, from which you draw approximately 1000 b.d. per well. What would be approximate draw down on a well of this nature?

MR MASTERSON: About 100 lb.

P. A. T. HAINES: I understand from the paper that the four sands in the Burgan field have a common level for the oil/water contact. In view of the fact that the four sands are separated by shales, can this common level be explained by faulting, or is it unique in that field?

MR MASTERSON: There is definitely a common oil/water contact level; there is probably direct connexion between the main bodies of producing sands. Faulting could have had an effect on this.

R. K. DICKIE: In the Burgan reservoir, as Mr Masterson has said, we believe that the original oil/water level in all sands was the same. In the first sand the oil/water level is not definitely known, but the original level was probably the same as in the other sands, and evidence from one well rather points in this direction. In the third and fourth sands there is very strong evidence that the original oil/water level was the same. This is not surprising, as there should be sufficient connexion between the various formations comprising the Burgan series, either directly or through faults for pressure equilibrium, to have established itself throughout the ages and, certainly between the third and fourth sand, even if some disequilibrium may now be caused by production. Even the first sand, which in the normal succession is divided from the other oil-bearing sands by the relatively impermeable Orbitolina limestone, must be in direct contact and so in original pressure equilibrium with the other sands through faulting.

H. R. Lovely : I should like to ask about the electric logging at Burgan. Is any attempt made to interpret the electric logs in a quantitative way to obtain water-porosity values and so on, and if so, is there a particular arrangement of electrode spacing ? Also, is the micro log run for selecting the perforation intervals ?

With reference to the coalescence of sand members to which reference has been made, I think we have a similar situation in our Zubair field (Basrah Petroleum Co.). In this field the first few wells showed the producing zone to be a sand and shale complete with, however, a definite shale bed in the middle of the section which could be followed from well to well and appeared to separate the reservoir into an upper and a lower portion. Subsequent drilling has shown that this shale bed tends to disappear in a westerly direction so that the two sand sections are coming together, and it may be that they are interconnected and form a common reservoir.

Mr Masterson : Attempts have been made to interpret the Burgan logs quantitatively, but so far we have had little success. Although the logs are satisfactory for qualitative work, it appears that there is some unknown factor appearing which invalidates the standard formulæ. The electrode spacing normally employed is 10 and 40 inches, but 16 and 64 inches have been used.

We do not employ the micro log over our productive series. It has been found that this technique gives little or no advantage over the standard log in the Burgan sand series and, although we have made use of it in special circumstances, our perforations are always selected from a 1 : 200 vertical scale electric log.

R. L. Messent : You have mentioned orifice meters on the incoming flow lines from the wells to the gathering centres. Since the incoming fluid is in two phases, *i.e.*, oil and gas, each of which will have diverse flow differential across the orifice, how is an accurate measurement obtained ?

Mr Masterson : We measure the mixture of oil and gas as it enters the gathering centre. The result is, of course, not very accurate ; the measurements can be up to 10 per cent off. As there are only fourteen to twenty wells feeding each gathering centre, we can run the production from an individual well through the test separators into a 10,000-brl measuring tank. This we do from time to time, thus obtaining an accurate figure of the production of any one well. We also have two 50,000-brl tanks at each centre, and knowing the total receipts in any 24-hr period, we allocate the production to each well in proportion to the figures we get from the meters, corrected where necessary in accordance with the figures obtained on actual well-production tests.

Professor G. I. Finch : My impression, after listening to this paper, is that we are left with one great outstanding problem, the disposal of the gas. In view of the quantities involved, carbon black, steam-raising, and so forth only nibble at the problem. It may be that re-pressurizing is the only solution until we have learned, through methane chemistry research, how to use the gas directly. It may also be that re-pressurizing may not

only store the gas for the future but also even return an immediate dividend by improving the quality of the oil.

MR MASTERSON : I heartily agree; we are very actively engaged in the study of that problem at the moment.

THE PRESIDENT : It is not an unknown problem; it occurs in other parts of the world also.

S. J. M. AULD : What is the approximate sulphur content of the separated gas? In my recollection of many years ago, in the Persian field it was about 10 per cent of H_2S. The Kuwait crude oil has the reputation of possessing a very high sulphur content, and I wonder if the gas is worthy of attention in respect of its sulphur content before you send it into the ground again. If it is similar to the Persian gas, and unless my calculation is wrong, it looks as though you might have 100,000 tons of sulphur available yearly; that is quite a bit, in view of the world shortage of sulphur at the present time.

MR MASTERSON : There is an important difference between the H_2S content of the gas in Persia and Kuwait. Whereas the content is quite appreciable in Persia, Kuwait gas contains only a trace. As a result, there can be no question of sulphur recovery from gas in Kuwait.

E. S. PINFOLD : I was very much interested in the point that the oil is under-saturated, and I think you said at one stage that you have a long way to go before experiencing gas troubles. What sort of gas troubles do you expect when you get to saturation point?

MR MASTERSON : The formation of a gas cap on the crest of the structure.

THE PRESIDENT : I should like to say again how much we are indebted to Mr Masterson. He has made the observation this evening that in a development of this kind there are many problems other than oil production and oil handling; I refer to the problems of housing, water supplies, sanitation, food, etc., all of a much more difficult nature than the actual oil problem. We are glad to see here Sir Godfrey Mitchell, who, with Mr Coutts and his team, helped us during the most difficult stage of the work by providing housing facilities for the men who were doing the job which Mr Masterson has talked about.

On behalf of everyone present, Mr Masterson, I want to say that we thank you very much; everyone will wish to join with me in expressing our thanks.

(The vote of thanks was heartily accorded.)

MR MASTERSON : Thank you very much, Mr President and gentlemen. I should like to take the opportunity to say that Mr Boaden has done a great deal of work in connexion with the preparation of this paper. This he did during his summer vacation from the Persian Gulf. I am very sorry that he is not here this evening to see how much you appear to have enjoyed our joint effort.

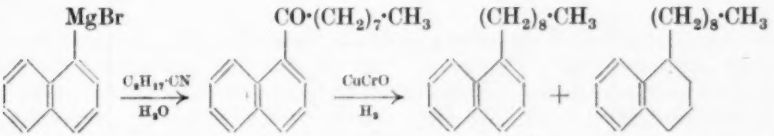
ALKYLNAPHTHALENES

PART VII. THE PHYSICAL PROPERTIES OF *n*-ALKYLNAPHTHALENES

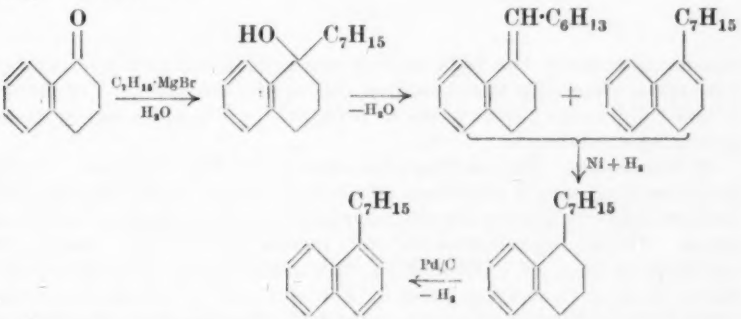
By D. G. Anderson and J. C. Smith (Fellow)

In Part III [1] of this series the preparation and the physical properties of the lower alkylnaphthalenes were described. The present paper deals with members having side-chains of 7 to 11 carbon atoms; in these compounds the physical properties resemble those of aliphatic rather than of aromatic hydrocarbons.

As in Part III the β-alkyl compounds were most conveniently made through the β-naphthyl-*n*-alkyl ketones obtained by the Friedel–Crafts reaction. For the α-compounds from α-*n*-butyl to α-*n*-hexyl the Wurtz–Fittig reaction with α-bromonaphthalene was preferred, but for the higher homologues condensation of α-naphthylmagnesium bromide and an alkyl-cyanide [2] has given (as in a parallel investigation in this laboratory [3]) high yields of ketones. Reduction of these ketones with hydrogen and copper chromite produced the alkylnaphthalene (usually contaminated with nuclear reduction products). Purification by catalytic dehydrogenation, followed by crystallization from solvents, yielded satisfactory products.



In the case of α-*n*-heptylnaphthalene a different route was used, that through α-tetralone :—



The mixture of isomers produced by the dehydration of the carbinol would probably have been difficult to dehydrogenate (as in the corresponding α-*n*-hexyl-derivative [1, p. 113]), so first it was reduced to the homogeneous α-*n*-heptyltetralin. This route was later abandoned in favour of the one using the nitrile and ketone.

As would be expected from the experience of Part III, where the *n*-hexylnaphthalenes formed very unstable molecular compounds, there was no sign in these more aliphatic homologues of ability to combine even with *sym*-trinitrobenzene.

Ultra-violet and infra-red spectra of these preparations have been obtained in several laboratories. The data here included are those kindly supplied by Mrs M. Broomfield and Miss E. Mikiewicz of the Physical Chemistry Group of I.C.I. Ltd., Billingham Division.

It is hoped later to publish a comprehensive paper on the spectra of these compounds, and in particular to include the infra-red spectra taken on a double-beam instrument.

Boiling points. With the apparatus available it has not been possible to

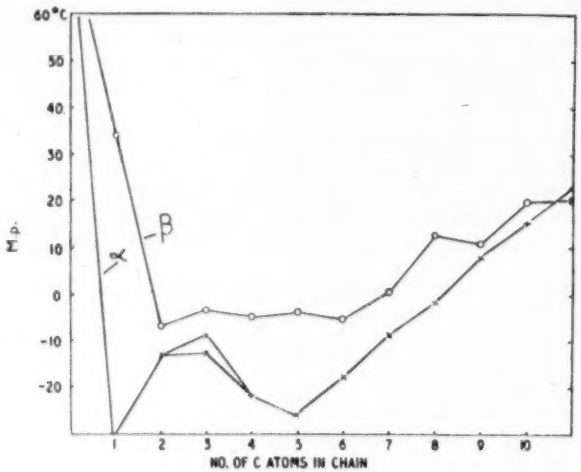


FIG 1

measure accurately the high boiling points of these new hydrocarbons; it would be interesting to see whether the curves for *b.p./no. of C atoms* of the α- and the β-series (which cross at *n*-propyl,[1, p. 116]) approach again with increasing chain-length.

Melting points. The melting-point curves call for comment. In Fig 1 it is seen that with a side-chain of about 6 carbon atoms the alkylnaphthalenes begin to take on the characteristics of purely aliphatic homologous series. But the behaviour of the early members is striking: starting from naphthalene (m.p. 80°) introduction of a methyl group into the α-position causes a drop in melting point of 111° (to −31°); in the β-position a methyl group depresses the m.p. only 46°. (Introduction of methyl into benzene depresses the m.p. by 96°.) This large depression with the α-methyl group causes α-methylnaphthalene to be the lowest-melting of all the *n*-alkylnaphthalenes. In most homologous series methyl homologues are the highest-melting of the early members, and then there is an irregular fall. The second minimum (at α-C_5) is met in many aliphatic series at *n*-amyl

(often due to the use of impure amyl halides !). From the general narrowness of the m.p. range for the C_2 to C_6 homologues, both α- and β-, it is inferred that there is a fairly constant symmetry or crystallographic factor.

When the side-chain (α- or β-) has six or more carbon atoms the m.p/

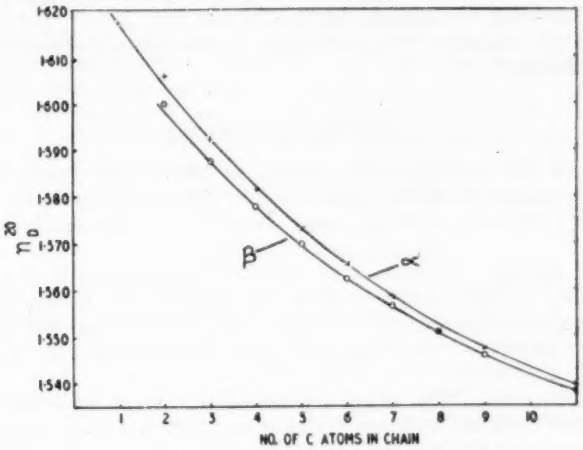


FIG 2

carbon curves resemble those of a purely aliphatic homologous series, the α-curve being less inflected for the odd and even members than the β-curve. Certainly as far as C_{10} the β-compounds are higher-melting than the α-isomers. In Fig 1 the data for the octyl- and the decylnaphthalenes are those given by Bannister and Elsner.[3]

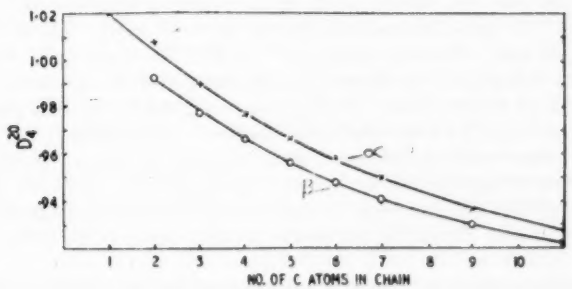


FIG 3

Refractive indices. From the experience obtained in purifying these compounds it seems that the refractive index is a more useful criterion of purity than is the m.p. (unless the latter is taken with special care). A small amount of ketone or of tetrahydronaphthalene is easily detected by a rise or fall, respectively, of the refractive index. Conversely, in comparing

the refractive index/carbon curves for the α- and β-series it is essential to use pure materials.

In Fig 2 the data for the octylnaphthalenes (α- and β-, 1·5510 and 1·5511, respectively) are from Bannister and Elsner's paper.[3]

Densities. The two density/carbon curves gradually approach, the α-compounds being the denser (Fig 3).

Data on the members from *n*-dodecyl- to *n*-hexadecylnaphthalenes are being accumulated.

1-*n*-Heptylnaphthalene

α-Tetralone was prepared from benzene by the usual succinic anhydride synthesis[4] except that the reduction of β-benzoylpropionic acid was conveniently effected in 83 per cent yield by the Huang-Minlon[5] procedure.

n-Heptyl alcohol. A solution of distilled *n*-heptaldehyde (100 g, b.p. 154° to 156°) in ethyl alcohol (250 cc) was shaken with Raney nickel and hydrogen at 115 atm and 150° for 8 hr. The resulting alcohol (b.p. 176°, n_D^{20} 1·4250; literature: 176° and n_D^{20} 1·425) was obtained in 94 per cent yield.

n-Heptyl bromide. The alcohol (63·8 g, 0·55 mol) hydrobromic acid (240 g or 160 cc of 48 per cent, 1·4 mol) and conc sulphuric acid (62 g or 34 cc) were heated 6 hr under reflux. From the mixture 91 g (92 per cent yield) of colourless *n*-heptyl bromide (b.p. 66·5° to 67°/19 mm, n_D^{20} 1·4508) were obtained. Vogel[6] gives b.p. 177·5°/758 mm and n_D^{20} 1·4508.

1-*n-Heptyl*-3 : 4-*dihydronaphthalene* (and isomer). A Grignard reagent was prepared from magnesium (6·1 g, 0·26 atom), *n*-heptyl-bromide (45 g, 0·25 mol) and ether (80 cc). To this a solution of freshly distilled α-tetralone (29 g, 0·20 mol) in dry ether (50 cc) was added with stirring during 1·5 hr, the clear solution left overnight at room temperature and then refluxed for 20 min. The solution yielded 50 g of crude carbinol, which, dehydrated for 1 hr with 98 per cent formic acid (80 cc), gave a mixture of olefins b.p. 150° to 184°/15 mm (but mainly 174° to 178°/15 mm). On redistillation the 18 cc of main fraction (b.p. 175° to 177°/15 mm) gave the correct analysis for *n*-heptyl-3 : 4-dihydronaphthalene and its isomers. (Found: C, 89·45; H, 10·60 per cent. $C_{17}H_{24}$ requires C, 89·5; H, 10·5 per cent.)

5-*n-Heptyl*-1 : 2 : 3 : 4-*tetrahydronaphthalene.* The olefin (13 g) in dioxan (50 cc), shaken with hydrogen (initial pressure 57 p.s.i.) in presence of Raney nickel (3 g) for 5·5 hr at room temperature, gave an oil of b.p. 172° to 173°/15 mm and n_D^{20} 1·5140, stable to permanganate solution. (Found: C, 88·6; H, 11·15 per cent. $C_{17}H_{26}$ requires C, 88·7; H, 11·25 per cent.)

1-n-*Heptylnaphthalene.* The *n*-heptyltetrahydronaphthalene (6 g) was dehydrogenated by heating with palladized charcoal (20 per cent Pd) in a small Claisen flask at 270° for 6 hr. During the last 2 hr nitrogen was bubbled slowly through the liquid. The product, recovered with the aid of light petroleum (b.p. 40° to 60°) and twice distilled (b.p. 140° to 141·5°/0·5 mm and 180°/15 mm), was a colourless oil (4·5 g; m.p. −9° to −8° and n_D^{20} 1·5577). In order to remove any trace of impurity (ketonic, olefinic, or carbinol) the liquid was heated 1 hr at 170° and stirred with sodium,

air being excluded. Only a slight yellowing was observed. Distillation gave four fractions:—

(1) b.p. 131°/0·5 mm, n_D^{20} 1·5558;
(2) b.p. 117°/0·002 mm, n_D^{20} 1·5579 m.p. −8·6°;
(3) b.p. 117°/0·002 mm, n_D^{20} 1·5579 m.p. −8·4°;
(4) b.p. 117° to 120°/0·002 mm, n_D^{20} 1·5580.

Fraction 2, crystallized from acetone at −30°, gave a product of m.p. −8·2°, n_D^{20} 1·5583; fraction 3 also gave a product of m.p. −8·2°, n_D^{20} 1·5583; fraction 1 was purified to m.p. −8·8° and n_D^{20} 1·5571. *Pure* 1-n-*heptylnaphthalene*: the products of m.p. −8·2° recrystallized from acetone still melted at −8·2° and had n_D^{20} 1·5583; D_4^{20} 0·9500. (Found: C, 90·3; H, 9·7 per cent. $C_{17}H_{22}$ requires C, 90·3; H, 9·7 per cent.)

2-*n*-Heptylnaphthalene

n-*Heptoic acid*. Following the procedure of Diaper,[7] freshly distilled *n*-heptaldehyde (124 g, b.p. 154° to 156°) was added to a solution of pure manganese sulphate (100 g) in water (200 cc) contained in a 500-cc flask fitted with a mercury-seal stirrer, an air-inlet reaching to the bottom of the flask, and a reflux condenser, the top of which was connected to a water-pump. While the mixture was *rapidly* stirred air was drawn through the liquid for 47 hr. The organic layer was separated, the aqueous layer extracted with ether, and the ether extract added to the organic layer. After washing with dilute sulphur dioxide solution, this organic layer was twice extracted with 2*N*-sodium carbonate solution to remove the heptoic acid. From the alkaline extract *n*-heptoic acid was obtained as an oil of b.p. 110° to 113°/18 mm, f.p. −9° and n_D^{20} 1·4249. The yield was 74 g (52 per cent), and 22 g of *n*-heptaldehyde were recovered. In the literature *n*-heptoic acid has b.p. 222°/764 mm, m.p. −9° to −7·5°; n_D^{20} 1·423 to 1·425. By the action of thionyl chloride the acid was converted in 80 per cent yield into *n*-heptoyl chloride of b.p. 62·5°/18 mm, n_D^{15} 1·4344, n_D^{20} 1·4320.

2-*Naphthyl*-n-*hexyl ketone*. *n*-Heptoyl chloride (64 g, 0·43 mol) was added to a solution of naphthalene (56 g, 0·43 mol) in 180 cc of dry nitrobenzene. (The commercial nitrobenzene was steam-distilled and then dried with calcium chloride for one week.) The mixture was cooled in an ice-bath and stirred while finely powdered anhydrous aluminium chloride (59 g, 0·44 mol) was added gradually during 50 min and the temperature maintained below 0°. After having stood overnight at room temperature the mixture was poured on to ice and dilute hydrochloric acid (with stirring). It was left 1 hr, transferred to a separating funnel with the aid of ether, washed with hydrochloric acid, with water, and then dried with calcium chloride. Distillation *in vacuo* gave a colourless oil (b.p. 170°/0·1 mm), which solidified (m.p. 58° to 60°; yield 63·5 g or 61 per cent, calc on the acid chloride). On crystallization from ethyl alcohol the substance separated as colourless plates, m.p. 59·5° to 60·5°. Buu-Hoï and Cagniant [8] gives m.p. 60°. (Found: C, 84·8; H, 8·3 per cent. Calc for $C_{17}H_{20}O$, C, 85·0; H, 8·3 per cent.) The 2:4-dinitrophenylhydrazone, small orange needles from alcohol, melted at 186°. (Found: N, 13·4 per cent. $C_{23}H_{24}O_4N_4$ requires N, 13·3 per cent.)

Proof of the Constitution of the Ketone. Following the method used by Bailey, Pickering, and Smith, [1, p. 115] the ketone in dioxan solution was oxidized with selenium dioxide, and the resulting α-diketone was further oxidized with alkaline hydrogen peroxide to yield β-naphthoic acid of m.p. 182°, identical with a synthetic specimen.

2-n-*Heptylnaphthalene.* 2-Naphthyl-*n*-hexyl ketone (45 g) and hydrazine hydrate (20 cc of 90 per cent) were added to a solution of sodium (10 g) in diethylene glycol [5] (280 cc) and the mixture refluxed 1½ hr to form the red hydrazone. After removal of the condenser and heating until the mixture boiled at 200° the liquid was kept refluxing for 4 hr, becoming almost colourless but with a violet fluorescence. The resulting crude, oily heptylnaphthalene gave, on distillation, 35 cc of a main fraction of b.p. 140° to 150°/0·5 mm. Redistillation of this gave 23 g of b.p. 145° to 148°/0·5 mm, n_D^{20} 1·5568, and m.p. 0·2°.

Two treatments with sodium at 170° (stirring for ½ hr each time and then distilling *in vacuo*) gave :—

	n_D^{20}	D_4^{20}	M.p.
Fraction (i) . . .	1·5562	0·9409	0·5°
„ (ii) . . .	1·5565	0·9407	0·6°
„ (iii) . . .	0·5562	0·9404	0·5°

Fraction (ii) was taken as the purest sample and analysed. (Found : C, 90·1 ; H, 9·6 per cent. $C_{17}H_{22}$ requires C, 90·3 ; H, 9·7 per cent.)

1-*n*-Nonylnaphthalene

Following the procedure of *Organic Syntheses* [9] a sample of pure *n*-octyl alcohol was converted in 95 per cent yield into *n*-octyl bromide, b.p. 91° to 93°/22 mm and n_D^{25} 1·4503, n_D^{20} 1·4525. Vogel [6] gives n_D^{20} 1·45267.

Pelargononitrile (*n*-Octyl cyanide). A solution of pure potassium cyanide (29 g, 0·44 mol) in water (60 cc) and ethyl alcohol (150 cc) was added to a solution of *n*-octyl bromide (64 g, 0·33 mol), and the mixture was refluxed for 8 hr in an oil-bath kept at 150° to 160°. Removal of the alcohol (by distillation) and filtration from potassium bromide left two liquid layers. Ether was added, and the ethereal layer, after washing with water and drying, yielded 53 g (86 per cent) of a colourless oil b.p. 100° to 101°/19 mm, n_D^{20} 1·4255, n_D^{25} 1·4235. (Literature values, n_D^{20} 1·4252, n_D^{25} 1·4235.[10])

1-*Naphthyl*-n-*octyl ketone.* The α-bromonaphthalene was purified by the method of Jones and Lapworth [11] and melted at +5·8°. To a mixture of magnesium turnings (3·6 g, 0·15 atom) and ether (25 cc) a drop of bromine was added and then, gradually, a solution of pure α-bromonaphthalene (30 g, 0·14 mol) in ether (40 cc), while the mixture was stirred and the flask heated gently. When only a trace of magnesium remained a solution of pelargononitrile (19 g, 0·14 mol) in ether 40 cc was slowly run in, the mixture being stirred and heated so that it refluxed gently. Heating was continued for 6 hr and the product then isolated. After naphthalene (4 g) had distilled the 1-naphthyl-*n*-octyl ketone passed over as a yellow oil, b.p. 170° to 171°/0·2 mm. (Yield 24 g, or 65 per cent, based on the nitrile.) The oil had m.p. −8° and n_D^{20} 1·5580. (Found : C, 85·1 ; H, 8·9 per cent. $C_{19}H_{24}O$ requires C, 85·1 ; H, 8·9 per cent.)

Attempts to make the semicarbazone or dinitrophenylhydrazone failed,

as with other α-naphthyl alkyl ketones, [see 1, p. 112] and the CO group could not be reduced by the Huang-Minlon [5] method.

5-n-*Nonyl*-1 : 2 : 3 : 4-*tetrahydronaphthalene*. 1-Naphthyl-*n*-octyl ketone (26 g) was stirred in a hydrogenator with hydrogen at an initial pressure of 100 atm in the presence of copper chromite catalyst (5 g) for 24 hr at 200° to 230°. The product, isolated with the aid of petroleum ether (b.p. 60° to 80°), was a yellow oil of b.p. 190° to 192°/1·5 mm. Heating with sodium metal to 150° and then distillation (b.p. 140° to 142°/0·01 mm) gave 13 g of colourless oil, n_D^{20} 1·5103. (Found : C, 88·4; H, 11·8 per cent. $C_{19}H_{30}$ requires C, 88·4; H, 11·6 per cent.)

1-n-*Nonylnaphthalene*. 5-*n*-Nonyltetrahydronaphthalene (13 g) dehydrogenated under the same conditions as used with 5-*n*-heptyltetrahydronaphthalene gave 10 g of liquid, b.p. 150°/0·1 mm and n_D^{20} 1·5461. This product, stirred with sodium at 170° for 0·5 hr (in order to remove ketonic impurities) and then distilled from a Claisen flask, gave a main fraction of b.p. 137° to 139°/0·02 mm and m.p. 7·6°, n_D^{20} 1·5467. Fractional distillation with a micro-column gave three fractions : (i) b.p. 137° to 138°/0·05 mm, n_D^{20} 1·5460; (ii) b.p. 138° to 139°/0·05 mm, n_D^{20} 1·5468 and m.p. 7·8°; (iii) b.p. 138° to 139°/0·05 mm, n_D^{20} 1·5479 and m.p. 7·5°. The main fraction (ii), crystallized from acetone at −20°, gave good crystals of a product melting sharply at 8·0° and having n_D^{20} 1·5472, D_4^{20} 0·9367. (Found : C, 89·6; H, 10·1 per cent. $C_{19}H_{26}$ requires C, 89·8; H, 10·2 per cent.)

2-*n*-Nonylnaphthalene

Pelargonic acid. A mixture of pelargononitrile (100 g), potassium hydroxide (88 g), water (60 cc), and ethyl alcohol (220 cc) was refluxed for 66 hr. The resulting pelargonic acid (97 g, 85 per cent yield) was a colourless oil of b.p. 138° to 140°/18 mm and n_D^{20} 1·432. (Literature,[12] b.p. 154°/760 mm, n_D^{20} 1·431.)

Pelargonyl chloride was obtained in 90 per cent yield by the action of thionyl chloride, and boiled at 96° to 98°/15 mm.[13]

2-*Naphthyl*-n-*octyl ketone* was prepared in the same manner as 2-naphthyl-*n*-hexyl ketone by adding anhydrous aluminium chloride (68 g, 0·51 mol) to a solution of naphthalene (66 g, 0·5 mol) and pelargonyl chloride (91 g, 0·51 mol) in pure dry nitrobenzene (450 cc). The yield of crude ketone, b.p. 170° to 174°/0·1 mm, was 101 g (73 per cent). Crystallization from alcohol gave 84 g of large colourless plates, m.p. 57°. (Buu-Hoï and Cagniant [8] give m.p. 58°.)

The 2 : 4-dinitrophenylhydrazone separated from ethyl acetate in red plates of m.p. 145°. (Found : N, 12·45 per cent $C_{25}H_{28}O_4H_4$ requires N, 12·5 per cent.)

2-n-*Nonylnaphthalene*. The reduction of the ketone was carried out as for 2-*n*-heptylnaphthalene, using sodium (8·3 g) dissolved in diethylene glycol (210 cc), 2-naphthyl-*n*-octyl ketone (40 g), and hydrazine hydrate (15 cc of 90 per cent). In this case 4 hr were allowed for the hydrazone formation, then the temperature of the boiling liquid was raised to 230° (by allowing water to escape) and refluxing at 230° continued for 8 hr. The resulting brown oil, distilled at 160° to 165°/0·2 mm, gave a light yellow distillate. This was stirred with sodium at 150° to 160° for ¾ hr and then distilled,

giving a small fraction (i), b.p. 134° to 150°/0·08 mm, n_D^{20} 1·5466, and a main fraction (ii), b.p. 150 to 153°/0·08 mm, n_D^{20} 1·5463. This main fraction (25 g) was colourless, but it was again stirred with sodium at 170° for ½ hr and then distilled at 138° to 140°/0·08 mm; the n_D^{20} was 1·5465. On cooling in acetone at −20° it formed a soft mass which, after stirring, changed to hard crystals of m.p. 10·9° and n_D^{20} 1·5459. Again crystallized from acetone it melted at 11·0° with n_D^{20} 1·5460 and D_4^{20} 0·9304. (Found: C, 98·8; H, 10·05 per cent. $C_{19}H_{26}$ requires C, 89·8; H, 10·2 per cent.)

1-*n*-Undecylnaphthalene

n-*Decyl bromide*. *n*-Decyl alcohol (86 g, m.p. 5°) was added to a mixture of hydrobromic acid (480 g, or 320 cc, of 48 per cent) and conc sulphuric acid (124 g, 68 cc). The mixture, after having been refluxed for 6·5 hr, yielded 217 g (90 per cent) of *n*-decyl bromide, b.p. 106° to 107°/10 mm, n_D^{20} 1·4553. Vogel[6] gives n_D^{20} 1·45527.

Undecanonitrile. A solution of pure potassium cyanide (58 g, 0·89 mol) in water (120 cc), ethyl alcohol (300 cc), and *n*-decyl bromide (148 g, 0·71 mol) was refluxed for 8 hr and yielded 86 g (77 per cent) of undecanonitrile, b.p. 118° to 120°/15 mm, n_D^{20} 1·4333. (Literature, n_D^{20} 1·4330; Timmermans [10] gives n_D^{20} 1·43286.)

1-*Naphthyl*-n-*decyl ketone*. To the Grignard reagent prepared from α-bromonaphthalene (45 g, 0·22 mol), magnesium (5·4 g, 0·22 atom), and ether (100 cc) was added a solution of undecanonitrile (35·2 g, 0·21 mol) in ether (60 cc). From this mixture (as in the preparation of 1-naphthyl-*n*-octyl ketone) the ketone (39 g), of b.p. 172° to 174°/0·06 mm, was obtained. On redistillation this gave a main fraction (34 g, 54 per cent yield) of b.p. 172° to 174°/0·06 mm, n_D^{20} 1·5567. (Found: C, 85·4; H, 9·6 per cent. $C_{21}H_{28}O$ requires C, 85·1; H, 9·5 per cent.)

1-n-*Undecylnaphthalene*. The ketone (31 g) with copper chromite (7 g) and hydrogen initially at 115 atm, heated 8 hr and stirred at 250° to 280°, gave a product of b.p. approx 145°/0·005 mm and n_D^{20} 1·5343 to 1·5372 (throughout the distillation). As it was obvious that the nucleus also had suffered reduction, all the product was dehydrogenated with Pd/charcoal at 280° for 1 hr. Collected in five fractions the distillate had n_D^{20} 1·5372, 1·5382, 1·5386, 1·5394, and 1·5404 respectively. Fractions 1, 2, and 3, crystallized from acetone at −15°, gave a product of m.p. 22·5°, $n_D^{27·4}$ 1·5358, n_D^{25} 1·5370. From these, n_D^{20} (calc) was 1·5390. Fractions 4 and 5, crystallized from acetone, gave 2·8 g of m.p. 22·4°, n_D^{20} 1·5388. (This value for n_D^{20} was obtained on the supercooled liquid.) (Found: C, 89·3; H, 10·8 per cent. $C_{21}H_{30}$ requires C, 89·4; H, 10·6 per cent.) The main specimen of m.p. 22·5° was further purified chromatographically in the Koninklijke/Shell-Laboratorium, Amsterdam, and was then found to have m.p. 23·1° and n_D^{20} 1·5398 (Shell measurements) or m.p. 22·8°, n_D^{20} 1·5394, D_4^{20} 0·9280 (authors' measurements).

2-*n*-Undecylnaphthalene

n-*Undecoyl chloride*. A solution of undecenoic acid (280 g, m.p. 24·5°) in glacial acetic acid (300 cc), reduced with hydrogen at an initial pressure of 80 atm and at room temperature in presence of palladium/barium sulphate

for 7 hr, gave 240 g (85 per cent yield) of *n*-undecoic acid, b.p. 150°/12 mm and m.p. 29·5° to 30·5°.[14] The acid was stable to permanganate solution, even on long standing. By the action of thionyl chloride this acid (93 g) was converted to the chloride, b.p. 77° to 78°/0·1 mm, in a yield of 92 g (90 per cent).

2-*Naphthyl*-n-*decyl ketone*, obtained from the acid chloride, naphthalene, and aluminium chloride in pure, dry nitrobenzene, was a crystalline solid of m.p. 47° (55 per cent yield). Buu-Hoï and Cagniant[8] gives m.p. 48°. The 2 : 4-dinitrophenylhydrazone, red plates from ethyl acetate melted at 127°. (Found : N, 11·8 per cent. $C_{27}H_{32}O_4N_4$ requires N, 11·8 per cent.)

2-n-*Undecylnaphthalene*. The 2-naphthyl-*n*-decyl ketone was reduced by the Huang-Minlon procedure (4 hr for formation of the hydrazone and 7 hr at 230° for the reduction). The product, treated with sodium for ½ hr at 160° gave on distillation a small fraction of n_D^{20} 1·5385 and the main fraction of n_D^{20} 1·5388. Repetition of the sodium treatment gave a product of b.p. 151°/0·007 mm and n_D^{20} 1·5386. On cooling to 16° the liquid hydrocarbon set to a jelly and the temperature rose slowly. In an air-jacket at 20° with the thermometer in the melt the mass became a clear liquid at approximately 18·5°, If, on the other hand, the jelly-like mass was stirred vigorously with the thermometer it became opaque; after having stood for 24 hr the crystalline opaque mass melted at 19·5° to 20°. These results could be repeated.

Again the specimen was distilled through a small column : it boiled at 150° to 155°/0·005 mm (b.p. depending on speed of distillation) without any change in refractive index during fractionation. The main portion was recrystallized from acetone at −40° and separated from the solvent as hard opaque crystals. These were re-dissolved in acetone, recrystallized at −40°, and then all solvent removed under reduced pressure. The substance still set to a jelly on cooling, and this form disappeared between

TABLE I

n-Alkylnaphthalene	α-				β-			
	B.p., °C/mm	M.p.	n_D^{20}	D_4^{20}	B.p., °C/mm	M.p.	n_D^{20}	D_4^{20}
Heptyl	141/0·05 117/0·002	− 8·2 —	1·5583 —	0·9500 —	146/0·5 —	+ 0·6 —	1·5565 —	0·9407 —
Octyl	170/4 [8] 197/12 [15]	− 2 [8] —	1·5510 [8] 1·5532 [15]	— 0·9401 [15]	— 192·5/12 [15]	+13 [8] − 3·3 [15]	1·5511 [8] 1·5510 [15]	— 0·9390 [15]
Nonyl	139/0·05	+ 8·0	1·5472	0·9367	139/0·008	+11	1·5460	0·9304
Decyl	200/4 [8] —	+15 [8] —	1·5448 [8] —	— —	— —	+19·6 [8] +13 [16]	1·5440 [8] 1·5394 [16]	— —
Undecyl	145/0·005	+22·8	1·5394	0·9280	151/0·007	20·0	1·5382	0·9220

Data without superscripts are those obtained in this investigation.

18° and 19°, usually at 18·8°. Rubbing with a thermometer and leaving some hours produced a form of crystal clearing at 20·0°. It thus appears that, when supercooled, 2-*n*-undecylnaphthalene can exist as a liquid-crystalline form which is in meta-stable equilibrium with the supercooled, isotropic liquid at 18·8°; the solid crystalline form is in equilibrium

with this liquid at 20·0°. (Spectrographic examination showed that the specimen was entirely free from the α-isomer and from ketonic impurities. An earlier specimen of the hydrocarbon, which had become contaminated with the α-isomer, melted much less definitely and gave the α-lines plainly in the spectra.)

For the best specimen of 2-*n*-undecylnaphthalene the physical data are: m.p. 20·0°, n_D^{20} 1·5382, D_4^{20} 0·9220. (Found: C, 89·3; H, 10·8 per cent. $C_{21}H_{30}$ requires C, 89·4; H, 10·6 per cent.)

INFRA-RED SPECTRA OF ALKYLNAPHTHALENES

By (Mrs) M. Broomfield *

The infra-red spectra were recorded from 15 to 6 μ. All samples were liquids, in a cell of about 0·1 mm thickness. The spectrometer was a Hilger D.209 (used on single beam) with a prism of rock-salt.

In Table II are given the infra-red bands of some 1-*n*-alkylnaphthalenes,

TABLE II

Infra-red Spectra of 1-n-*Alkyl Naphthalenes* (15 *to* 6 μ)

Frequency (cm⁻¹)–Liquid			Relative intensity	Frequency (cm⁻¹)–Liquid			Relative intensity
1-*n*-heptyl	1-*n*-nonyl	1-*n*-undecyl		1-*n*-heptyl	1-*n*-nonyl	1-*n*-undecyl	
692	692	691	w	—	—	1123	w
726	721	721	s	1146	1145	1142	w
—	730	729	sh	1167	1166	1166	m
777	778	777	vs	1205	—	—	w
791	791	792	vs	1220	1218	1217	w
860	858	858	m	1241	1240	1241	w
891	889	888	w	1262	1262	1262	m
900	900	900	w	1300	1300	1300	w
949	948	948	m	1358	1358	1358	w
967	965	966	m	1378	1376	1378	m
1019	1016	1016	m	1396	1396	1396	m
1026	1026	1025	sh	1466	1466	1466	s
1081	1080	1079	m	1508	1508	1508	m
1117	1117	1115	w	1600	1600	1600	m

Note: s = strong; vs = very strong; m = medium; w = weak; sh = shoulder.

and in Table III the bands of the 2-*n*-alkyl isomers. It has been pointed out by Vago, Tanner, and Bryant [17] that great similarity is found among the spectra of the 1-substituted and of the 2-substituted naphthalenes with substituents ranging from methyl to *n*-hexyl. Comparison of the frequencies in Tables II and III shows that, as would be expected, the similarity of spectra of homologues is very marked in the series with longer-chain substituents; but the two types of substitution, α and β, can be readily distinguished by their infra-red spectra.

It is possible to correlate several bands of aromatic molecules with the structure (see *e.g.*, Thompson [18]). Aromatics are characterized by strong

* Physical Chemistry Group, I.C.I. Ltd., Billingham Division.

bands between 670 and 900 cm^{-1}, which are attributed to out-of-plane hydrogen-bending vibrations. These bands are correlated with substitution in the benzene ring, and may be extended to fit 1- and 2-substituted naphthalenes (Colthup [19]). Intense bands are present in the 1-substituted

TABLE III

Infra-red Spectra of 2-n-Alkyl Naphthalenes (15 to 6 μ)

Frequency (cm^{-1})–Liquid			Relative intensity	Frequency (cm^{-1})–Liquid			Relative intensity
2-*n*-heptyl	2-*n*-nonyl	2-*n*-undecyl		2-*n*-heptyl	2-*n*-nonyl	2-*n*-undecyl	
723	720	720	s	1173	1172	1171	w
745	745	745	vs	1212	1212	1212	w
814	814	815	vs	1246	1243	1244	w
855	854	854	s	1272	1270	1271	m
890	890	890	s	—	1304	1304	w
947	946	946	m	1376	1376	1376	m
959	959	959	m	1466	1466	1466	s
1020	1019	1019	m	1508	1508	1508	m
1127	1127	1127	m	1605	1605	1605	m
1146	1144	1144	w	1635	1635	1635	m
1157	1154	1155	w				

Note : s = strong; vs = very strong; m = medium; w = weak.

naphthalenes at 777 cm^{-1} and 792 cm^{-1}; in the 2-substituted naphthalenes at 750 cm^{-1} and 815 cm^{-1}. The lower frequency may be characteristic of the 1 : 2-disubstituted ring in these compounds and the higher of the 1 : 2 : 3- and 1 : 2 : 4-trisubstituted rings, respectively.

The well-known 1500- and 1600-cm^{-1} bands of aromatics, attributed to in-plane skeletal vibrations, are well marked in the 1- and 2-substituted naphthalenes. Another prominent feature of all the mono-*n*-alkylnaphthalenes is the " long-chain " band between 720 and 730 cm^{-1}, which is interpreted as a rocking mode of the CH_2 groups (Shepherd and Sutherland [20]).

ULTRA-VIOLET ABSORPTION SPECTRA OF MONO-ALKYL NAPHTHALENES

By (MISS) M. MIKIEWICZ *

The near ultra-violet absorption spectra of mono-alkyl naphthalenes consist of two regions of absorption. The short-wavelength region, from λ 2400 to 2950 Å is intense and composed of a series of rather widely spaced diffuse bands. Much more characteristic is the long-wavelength region extending from λ 2950 to 3250 Å, which, though much less intense, has a well-developed structure.

Table IV gives the wavelengths and molar extinction coefficients of bands of α and β series of *n*-heptyl- to *n*-undecyl-naphthalenes.

Within each series the spectra in general character strongly resemble

* Physical Chemistry Group, I.C.I. Ltd., Billingham Division.

each other and bear close resemblance to their α- and β-methyl homologues. As the table shows, the wavelengths of the bands remain constant within the experimental error of the measurements. There also appears to be no appreciable change in the intensity of the bands.

TABLE IV

Position and Intensity of Bands of C_7 to C_{11} n-Alkyl Naphthalenes

α-*n*-Heptyl		α-*n*-Nonyl		α-*n*-Undecyl	
λ (max)	Log ε	λ (max)	Log ε	λ (max)	Log ε
2535	3·34	2532	3·36	2532	3·31
2618	3·59	2620	3·62	2618	3·61
2719	3·84	2713	3·84	2720	3·83
2791	3·84	2788	3·86	2788	3·85
2832	3·93	2829	3·93	2832	3·94
2942	3·74	2944	3·76	2940	3·75
3036	2·99	3042	2·92	3032	2·93
3077	2·72	3076	2·65	3075	2·62
3102	2·68	3100	2·61	3103	2·56
3146	2·71	3146	2·65	3146	2·62
3183	2·11	3177	2·16	3184	2·11
3239	1·52	3237	1·76	3237	1·61
β-*n*-Heptyl		β-*n*-Nonyl		β-*n*-Undecyl	
λ (max)	Log ε	λ (max)	Log ε	λ (max)	Log ε
2561	3·63	2561	3·59	2557	3·59
2658	3·77	2658	3·60	2658	3·73
2765	3·82	2754	3·77	2755	3·79
2867	3·67	2864	3·59	2864	3·64
2980	2·71	2980	2·63	2975	2·67
3003	2·61	3003	2·51	3003	2·54
3050	2·76	3050	2·70	3051	2·75
3093	2·47	3091	2·41	3092	2·50
3117	2·49	3115	2·45	3117	2·54
3146	2·32	3142	2·27	3146	2·36
3192	2·73	3192	2·70	3195	2·75
3246	1·53	3248	1·58	3248	1·50

It is interesting to investigate what effect the increase of the length of the alkyl side-chain has on the position of the main bands. Table V collects the available data.

TABLE V

Comparison of the Position of Long-wavelength Maxima of α- and β-Monoalkyl Naphthalenes

	λ (max)		λ (max)
Naphthalene [22]	3110	Naphthalene [22]	3110
α-Methylnaphthalene [22]	3140	β-Methylnaphthalene [22]	3190
α-Ethylnaphthalene *	3141	β-Ethylnaphthalene *	3192
α-*n*-Heptylnaphthalene	3146	β-*n*-Heptylnaphthalene	3192
α-*n*-Nonylnaphthalene	3146	β-*n*-Nonylnaphthalene	3192
α-*n*-Undecylnaphthalene	3146	β-*n*-Undecylnaphthalene	3192

* Unpublished data.

Inspection of the table indicates that while the introduction of a methyl group into the α- or β-position of naphthalene produces considerable shift of the spectrum towards the red (in respect to naphthalene), the increase in the shift becomes progressively less with increasing length of the alkyl side-chain. It is difficult to judge from the incomplete table when the shift becomes constant, but it appears to be so above the *n*-heptyl group. There is no blurring of the structure with increasing length of the substituent, as has been noticed in the spectra of *n*-alkyl-substituted benzenes.[21]

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The Dyson Perrins Laboratory,
Oxford University.
September 12, 1951.

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INVESTIGATIONS ON THE DETERMINATION OF KINEMATIC VISCOSITY USING IP STANDARD METHODS

Introduction

This report deals with an experimental investigation carried out by members of the Viscosity Panel in their respective laboratories in an effort to provide data on the following subjects :—

1. The possibility of basing all viscometric measurements on a single value of viscosity as a primary standard, *i.e.*, the agreed value for the viscosity of pure water at 20·00° C.
2. The possibility of using master viscometers which could be based solely on the primary standard viscosity and which will not be subject either to kinetic-energy corrections or to surface-tension errors.
3. The use of master viscometers and present IP standard viscometers to evaluate oils in the viscosity range of 1 to 2000 cs by a stepping-up procedure.
4. The determination of the repeatability and reproducibility of viscosity determinations at different levels of viscosity values.

A brief summary of the work will be given followed by the main conclusions drawn and recommendations made as a result of the work. Appendices give :—

I. The specification of master viscometers used in the investigation.
II. Methods of using the master viscometers to determine the viscosity of a low viscosity hydrocarbon, *i.e.*, benzene.
III. Stepping-up procedures adopted.
IV. A summary of experimental results and their statistical analysis.

Summary of the Work

At a meeting of the Viscosity Panel held on March 6, 1946, it was decided to embark on the long-term investigation reported here. The statistical analysis was finished in March 1951. Of the five years, nearly a year was spent in designing the apparatus to the satisfaction of all participating members, approximately two years were lost in waiting for the manufacture of the apparatus, which was hindered by the prevalent post-war shortages of materials, and two years were utilized in the actual experimental and statistical work.

As a result of a detailed study of previous work carried out both in the U.K. and in the U.S.A., and with the help and advice of Dr G. Barr, who was the vice-chairman of the Panel until his retirement in 1950, viscometers were designed to be used as master instruments. The specifications of the two types—U-tube and suspended-level—are given in Appendix I, but a few explanatory notes will clarify the main criteria considered in the design.

Two types were designed in order to determine whether there was anything in favour of either from the viewpoint of accuracy. In both U-tube and suspended-level master viscometers the length and diameter of the tube were designed in conjunction with the capacity of the measuring-bulbs so that errors or corrections due to kinetic energy and to surface tension should be negligibly small. The internal diameters of the tubes where marks were placed were made small enough so that errors due to the finite width of the marks should be negligible. With the U-tube type it was deemed necessary to investigate whether filling the tube by the " Fenske method "—*i.e.*, inverting the U-tube with one limb in a beaker containing the sample and filling by applying suction at the other limb till the liquid filled a bulb to a standard mark—was a more accurate way of filling than that used with standard U-tubes, *i.e.*, with the viscometer in its upright position filling the lowest bulb of the viscometer to a standard mark. It was also desired to find whether accuracy was materially enhanced if the volume of the measuring-bulb for timing the flow was doubled. It was decided to design the U-tube master viscometer so that it could be used in several ways and thus gather the maximum information with the least amount of apparatus. Thus the U-tube was designed with two upper bulbs and one larger lower bulb. By tapering the entry of the tube leading to the upper bulb the viscometer could be inverted and filled by the Fenske method, using the lower of the two upper bulbs for timing. On the other hand, by filling the lower bulb, on the other limb, by means of a long pipette, the viscometer could be used as an ordinary Ostwald tube in three different ways : (*a*) the upper of the two bulbs could be used as a timing bulb; (*b*) the lower of the two bulbs could be used as a timing bulb; (*c*) both the upper and lower bulbs could be used as a single timing bulb with a total capacity of the order of twice the capacity of either. Finally, it was hoped that by comparing the results obtained with the U-tube type with those obtained with the suspended-level type the influence of surface tension would reveal itself; if results obtained by the two types used on, say, benzene agree to an acceptable degree, within that degree of accuracy surface-tension influences may be deemed as insignificant.

When agreement was obtained on the design of the viscometers, Mr G. F. Adams, of Messrs A. Gallenkamp and Co. Ltd., was approached and readily agreed to manufacture the viscometers and the bath to contain them. A tribute is due to him for his co-operation to produce both types of viscometers and a specially long bath to accommodate them and in which the temperature was controlled to the required accuracy of $\pm 0{\cdot}01°$ F.

The next step was to evolve a procedure for calibrating the master viscometers with water and to check the calibration by determining the viscosity of a typical low-viscosity hydrocarbon. The viscometers were calibrated according to the specification detailed in Appendix II, and a single batch of benzene was divided amongst the members for the viscosity determination.

At this stage the Viscosity Panel had the good fortune of having Dr G. F. P. Trubridge, of I.C.I. Ltd., join them as the statistician in charge of the analysis of the work. From this point onwards members transmitted all observations—*not* derived quantities, *e.g.*, means—to him, and he performed all mathematical operations on them. Thus the instructions

regarding obtaining standard deviations and means given in Appendix II were rescinded. To Dr Trubridge the Panel owe a debt of gratitude which it wishes to acknowledge.

After the work on benzene showed that both kinetic-energy and surface-tension effects were included in a coefficient of repeatability of 0·14 per cent and a coefficient of reproducibility of 0·25 per cent, it was decided to determine the viscosity of several oils in tubes of different denominations following as closely as possible the procedure laid down in IP 71/45(T). The interpretations of the instructions were purposely left to each laboratory in order to reveal any weakness in the method. Appendix III gives the brief instructions given to the participating laboratories on evaluating the viscosities of the oil samples circulated to them. Laboratories were free to choose their own tubes and their own stepping-up oils.

Finally, the results were again given to Dr Trubridge, whose analysis and conclusions are given in Appendix IV.

Conclusions

The Viscosity of Water

If the viscosity of water at 68·00° F (20·00° C) is taken as 1·0068 cs the mean values for the viscosity of water at 100·00° F and at 130·00° F come out at 0·6889 cs and 0·5185 cs with standard deviations of $\pm$0·00007 cs and $\pm$0·00008 cs respectively. The accepted values, from the literature, are 0·6891 cs and 0·5183 cs respectively with unknown weights.

In view of the doubt regarding the fourth figure in the viscosity of water at 100·00° F and 130·00° F, only the value of 1·0068 cs for water at 68·00° F should be adopted as a primary standard for viscometric work of the highest attainable accuracy. The values for 100·00° F and 130·00° F can be used only as check values when it is desired to test a master viscometer for non-significance of the kinetic energy and other secondary effects.

Master Viscometers

Of the types used—*i.e.*, the suspended-level and the U-tube types—and of all modes of filling the most repeatable and reproducible results were obtained with the U-tube type filled by a long pipette to a standard mark above the lower bulb and using the total capacity of the two upper bulbs for timing the flow. This is most probably due to the use of the larger volume of the two bulbs and not to any inherent superiority of the U-tube over the suspended-level method.

The viscosity of samples of low-cetane standard oil taken from one batch was determined at 68·00° F by both the U-tube and the suspended-level master viscometers, and the mean coefficient of reproducibility was found to be just below 0·2 per cent. It is concluded that surface-tension effects are not highly significant with the master viscometers used.

The kinetic-energy correction is not significant in the master viscometers used.

Master viscometers of the type and design used can have their calibration constants based solely on the primary standard viscosity of water at 68·00° F (20·00° C). The viscosity of water at 100° F and/or 130° F can then be used to establish the non-significance of the kinetic-energy correction.

Calibration and Stepping-up

If the viscosity of water at 68·00° F and a master viscometer with a timing bulb equal in capacity to the total of the two upper bulbs of the U-tube master viscometers used were adopted, then it would not be unduly strict to prescribe 200 sec as the minimum times of flow allowed for calibration purposes.

Kinetic-energy corrections for all viscometers used in the IP standard method have to be determined experimentally, and no guidance as to their magnitude can be given.

To evaluate the kinetic-energy term in the calibration of viscometers at least two oils or one oil at two temperatures should be used so that the times of flow are approximately in the two ranges 200 to 400 sec and 500 to 1000 sec respectively.

Repeatability and Reproducibility

The repeatability and reproducibility of viscosity determination by the methods followed in this investigation were found to depend on the number of calibration steps undertaken in reaching the viscosity level.

Estimated mean values for the repeatability, reproducibility, and mean reproducibility are given in Table 6.8 in Appendix IV. As an approximate summary, it may be generalized that :—

(*a*) repeatability is of the order of 0·14 per cent for the least viscous and 0·5 per cent for the most viscous oils tested;

(*b*) reproducibilities are 0·25 per cent and 1·50 per cent respectively.

Recommendations

It is recommended that :—

(1) IP 71/47(T) should be rewritten in the light of the work reported here and incorporating the use of master viscometers calibrated with water at 68·00° F (20·00° C). Calibration and stepping-up procedures, as well as expected repeatabilities and reproducibilities, should be amended to conform with the conclusions drawn from this work.

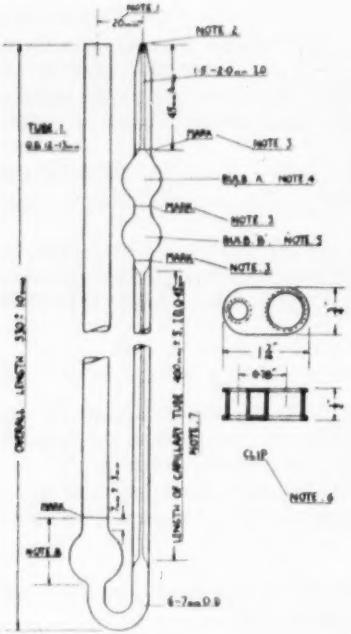
(2) The method so amended should be raised in status from "Tentative Standard" to "Standard."

Postscript

It was intended to investigate the influence of temperature up to 210° F on the calibration constants of standard viscometers, but the large amount of work involved precluded this further study.

Similarly, it has not proved possible to organize the regular testing and issuing of secondary standard oils in a manner similar to the issue of API oils in the U.S., nor the correlation of American and British viscometric methods.

These two items may well form the future programme of work for the Viscosity Panel, once the modified standard method is written and issued.



Note 1.—To fit clip.

Note 2.—Drawn to orifice 1 mm ID.

Note 3.—OD of tube at mark, 6–7 mm.

Notes 4 *and* 5.—Bulb capacity between marks, 2·5–2·6 ml; OD, 17–19 mm.

Note 6.—Clip cemented on tube with top $\frac{1}{2}$ inch from top of tube.

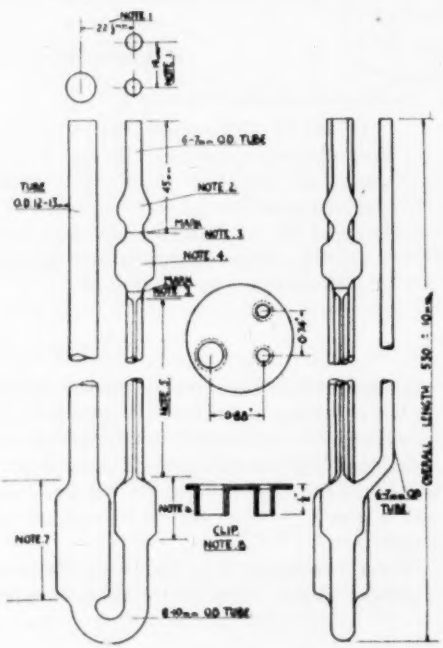
Note 7.—Capillary of precision bore tubing.

Note 8.—Capacity between mark and joint, $7 \pm 0{\cdot}5$ ml. Bulb 23–25 mm dia.

Material.—Heat resisting glass.

FIG 1

IP MASTER U-TUBE VISCOMETER



Note 1.—To fit clip.

Note 2.—OD of bulb, 12–14 mm.

Note 3.—OD of tube at mark, 6–7 mm.

Note 4.—Bulb capacity between marks, 2·5–2·6 ml.

Note 5.—Capillary, of precision bore tubing, 400 ± 5 mm long, 0·45 mm ID.

Note 6.—Bulb 17–19 mm OD, 25 ± 2 mm length.

Note 7.—Bulb 23–25 mm OD, 47 ± 3 mm length.

Note 8.—Clip cemented on tube with top $\frac{1}{2}$ inch from top of tube.

Material.—Heat resisting glass.

FIG 2

IP MASTER SUSPENDED-LEVEL VISCOMETER

Appendix I

DESIGN SPECIFICATIONS OF IP MASTER VISCOMETERS

There were two types of master viscometers designed :—

U-Tube Type (Fig 1)

The dimensions and tolerances were as shown. Further specifications were :—

A. The two limbs shall be parallel and straight.

B. The equatorial plane of the lowest bulb shall cut the lower end of the capillary tube at its junction with the wider tube.

C. Material of construction shall be as specified in "Standard Methods for Testing Petroleum and its Products," 1946, p. 465.

Suspended-level Type (Fig 2)

The dimensions and tolerances were as shown. Further specifications were :—

A. The three limbs shall be parallel and straight.

B. The material of construction as specified for the U-tube type.

Appendix II

THE CALIBRATION OF IP MASTER VISCOMETERS AND THE DETERMINATION OF THE VISCOSITY OF A BENZENE

Viscometer

The new IP viscometers have been constructed to the specifications in Appendix I. They are to be calibrated using pure water at 68° F, 100° F, and 130° F, and used to determine the viscosity of pure benzene samples also at 68° F, 100° F, and 130° F for stepping-up procedures.

Water-bath

The provision of a suitable constant-temperature water-bath is left to the discretion of each laboratory. A suitable bath must be about 50 cm deep in order to accommodate the viscometers, and must control the temperature within $\pm 0{\cdot}01$° F.

Thermometer

Only standard thermometers NPL certified to $\pm 0{\cdot}01$° F are to be used. The thermometer shall be calibrated not more than three months before testing.

Timing Device

Stop watches should be calibrated within three months both before and after tests to an accuracy of $\pm 0{\cdot}1$ per cent and the maximum allowable

variation between each calibration is to be $\pm 0{\cdot}1$ per cent. The escapement of the watch is arbitrary so long as these conditions are fulfilled. Electrical stop watches operated by synchronous motors which will be affected by voltage variations are not to be used unless steps are taken to prevent such variations. Stop watches should not be sent through the post.

Summary of Procedure for Calibration by Water

1. Drain; wash with chromic acid. Strength of acid and time of soaking arbitrary.

2. Drain; wash with ordinary distilled water to neutral, then wash once with triple-distilled water. Water to be considered neutral when pH 6·3 to 7·3 is attained (method of testing pH arbitrary) *and* when H_2SO_4 cannot be detected by the $BaCl_2$ test.

Allow to drain for $\frac{1}{2}$ hr after visible drainage ceases.

3. Fill by Fenske method and determine K_1 and A_1 in $\eta = K_1T - (A_1/T)$ from water at 68° F, 100° F, and 130° F; use five fillings and take triplicate timing readings for each filling, but discard the first timing reading for each filling.

4. Repeat cleaning procedure under (1) and (2) above.

5. Fill lowest bulb to mark without wetting tube above mark, use uppermost bulb as timing bulb and determine K_2 and A_2 as in (3) above.

6. Repeat cleaning procedure under (1) and (2) above.

7. Fill lowest bulb to mark, use lower of upper two bulbs as timing bulb, and determine K_3 and A_3 as in (3) above.

8. Repeat cleaning procedure under (1) and (2) above.

9. Fill lowest bulb to mark, use sum of both upper bulbs as timing bulb, and determine K_4 and A_4 as in (3) above.

10. Present results thus :—

Temperature, ° F	68·00	100·00	130·00
Average of ten times of flow, sec . . .	...	...	...
Standard deviation σ, sec . . .	...	...	...

$\sigma = [\frac{1}{9}\Sigma\,(\text{mean-reading})^2]^{\frac{1}{2}}$ $\quad K_1 =$ $\quad A_1 =$

Four viscometric constants will be available for the one instrument; the relative values of the four constants can be determined from their standard deviation.

Important Notes on Use of Water

1. Samples of triple-distilled water are to be obtained from B.D.H., and are to be used without any attempt at further purification.*

2. Tests should be carried out on the same day as cleaning.

3. The apparatus should be cleaned between each test and after standing overnight.

4. The apparatus should be left full of either water or chromic acid overnight.

* These samples were prepared from one large batch and sealed in glass ampoules. Later, laboratories had to prepare their own samples.

5. All glassware used in transferring liquids to the viscometer must be subjected to the same rigorous cleaning procedure as the viscometer itself.

6. Filtration of the water into the viscometer through sintered glass is to be arbitrary and is left to the discretion of the observer.

7. The maximum allowable temperature variation is to be $\pm 0{\cdot}01^\circ$ F or the limits of accuracy of the IP certified thermometer at the temperature of measurement, whichever is the greater.

8. Times of flow are to be measured to $\pm 0{\cdot}1$ sec.

9. Should any one method of filling and testing be found to be obviously inaccurate in comparison with the other three, then the results of this method should be discarded, and the use of this method should be discontinued.

Summary of Procedure for Determining Viscosity of Benzene

1. Drain; wash with chromic acid. Strength of acid and time of soaking arbitrary.

2. Drain; wash with water to neutral. Water to be considered neutral when pH 6·3 to 7·3 is attained (method of testing pH arbitrary) *and* when H_2SO_4 cannot be detected by the $BaCl_2$ test.

3. Drain; wash with redistilled acetone.

4. Drain; dry using dry, filtered air.

5. Wash with benzene.

Allow to drain for $\frac{1}{2}$ hr after visible drainage ceases.

6. Fill by Fenske method and determine η'_1 from $\eta'_1 = K_1T - (A_1/T)$ at 68° F, 100° F, and 130° F, using the values of K_1 and A_1 obtained by calibration with water; use five fillings and take triplicate timing readings for each filling, but discard the first timing reading for each filling.

7. Repeat cleaning procedure under (1) to (5) above (but see Notes (3) to (5) below).

8. Fill lowest bulb to mark, use uppermost bulb as timing bulb, and determine η'_2 as in (6) above.

9. Repeat cleaning procedure under (1) to (5) above (but see notes (3) to (5) below).

10. Fill lowest bulb to mark, use lower of upper two bulbs as timing bulb, and determine η'_3 as in (6) above.

11. Repeat cleaning procedure under (1) to (5) above (but see notes (3) to (5) below).

12. Fill lowest bulb to mark, use sum of both upper bulbs as timing bulb and determine η'_4 as in (6) above.

13. Present results thus :—

Temperature, ° F	68·00	100·00	130·00
Average of ten times of flow, sec .	...	...	...
Standard deviation σ, sec . .	...	...	...

$\sigma = [\frac{1}{9}\Sigma\ (\text{mean-reading})^2]^{\frac{1}{2}}$ $\eta' =$

Four values of viscosity will be determined for each temperature, corresponding to the four values of K and A determined by calibration with water.

Important Notes on Use of Benzene

1. Specially purified benzene samples * are to be obtained from the B.D.H.† sealed in 25-cc ampoules, and are to be used without any attempt at further purification.

2. Special precautions are to be taken to see that once the ampoules are opened the benzene is stored in contact with dry air, and then only for a short time.

3. Tests should be carried out on the same day as cleaning.

4. There should be no cleaning between tests carried out on the same day.

5. The apparatus should be cleaned after standing overnight using cleaning procedure under (1) to (5).

6. The apparatus should be left full of either benzene or chromic acid overnight.

7. All glassware used in transferring liquids to the viscometer must be subjected to the same rigorous cleaning procedure as the viscometer itself.

8. The maximum allowable temperature variation is to be $\pm 0{\cdot}01°$ F or the limits of accuracy of the IP certified thermometer at the temperature of measurement, whichever is the greater.

9. Times of flow are to be measured $\pm 0{\cdot}1$ sec.

Amendments to Instructions Given Above

1. In order to conserve samples of water and benzene a single filling should be used to time the double bulb and each of the single bulbs, allowing $\frac{1}{2}$ hr from drainage of upper bulb before lower bulb is used.

2. If original sample of water is exhausted redistilled, distilled water should be used.

3. In view of Dr Trubridge's offer to perform all statistical analysis, only observed times are to be reported and *not* means. It is not necessary to work out standard deviations or calculate values for K, A, or η'.

Appendix III

STEPPING-UP PROCEDURE AND DETERMINATION OF THE VISCOSITY OF OIL SAMPLES CIRCULATED TO LABORATORIES

It was agreed that each laboratory should interpret IP 71/45(T) for the stepping-up and use it to calibrate its own tubes by means of the IP Master viscometers already calibrated with water. The following special notes were to be observed by all laboratories :—

1. With each oil five fillings with duplicate readings on each filling shall be used.

* These were prepared from one large batch.

† Later, samples were provided by the Research Department, The National Benzole Co. Ltd., Watford, and circulated to the members for testing.

2. All tubes shall be stepped-up at the two temperatures 68° F and 130° F.

3. Each laboratory shall use oils from its own stocks for stepping-up the tubes. There is no need to use the same oil both at 68° F and 130° F.

4. The cetane standards circulated shall be tested in tube No. 1. Oil No. 1 shall be tested in tube No. 2. Oil PD 542 shall be tested in tube No. 3 and oil PD 543 in No. 4. The times of flow of these oils shall be observed at 68° F in tubes which have been previously calibrated by stepping-up from the master viscometer.

5. All times of flow, both for stepping-up and for measuring the viscosities of the special oils shall be returned to Dr Trubridge.

Appendix IV

STATISTICAL ANALYSIS OF EXPERIMENTAL RESULTS

Several hundred results and derived values were treated statistically by Dr G. F. P. Trubridge, and his conclusions were embodied in a report in six sections. This report is too voluminous to reproduce here, and consequently only Section 6, a discussion of the vast number of data contained in the first five sections is reproduced. The whole work, *in extenso*, is filed with the Institute of Petroleum for reference. To facilitate comparison between results reported herein and those filed at the Institute, paragraphs and tables are given numbers here which are identical with those in the full report, *e.g.*, tables are 6.1, 6.2, etc. Furthermore, references to earlier sections are left to indicate the basis for conclusions drawn in Section 6.

In the statistical analysis and tables the following notations are used :—

Laboratory No.	
1	Anglo-Iranian Oil Co. Ltd. (Sunbury-on-Thames).
2	Royal Aircraft Establishment (Farnborough).
3	Admiralty Chemical Department (Portsmouth).
4	" Shell " Refining and Marketing Co. Ltd. (Fulham).
5	Chemical Engineering Department of the University of Birmingham.
6	Aeronautical Inspection Department (Harefield).
7	Esso Development Co. Ltd. (Abingdon).
8	National Physical Laboratory (Teddington).
9	Koninklijke Shell-Laboratorium, Amsterdam.
10	Manchester Oil Refinery Ltd. (Manchester).
11	National Benzole Co. Ltd. (Willesden).

More than one set of results were obtained in some laboratories, while the programme was not completed in laboratories 2, 3, 5, and 8.

Notation

η	kinematic viscosity.
$\bar{\eta}$	mean kinematic viscosity obtained by one operator using one method at one temperature.
$\bar{\bar{\eta}}$	overall mean kinematic viscosity obtained by all operators using one method at one temperature.
T	time of flow.
$\bar{T}$	mean time of flow obtained by one operator using one method at one temperature.

Notation

K, A calibration constants.

$s(T)$ standard deviation of T.

$v(T)$ coefficient of variation of $T = \dfrac{s(T)}{T} \times 10^2$.

$s(K)$ standard deviation of K.

$v(K)$ coefficient of variation of $K = \dfrac{s(K)}{K} \times 10^2$.

$s(\eta)$ standard deviation of η.

$s(\bar{\eta})$ standard deviation of $\bar{\eta}$.

m, n number of observations.

N number of degrees of freedom.

$R_r(\eta)$ repeatability of η, *i.e.* the difference of samples of two values of η obtained by one operator using one set of apparatus.

$r_r(\eta)$ coefficient of repeatability of $\eta = \dfrac{R_r(\eta)}{\bar{\eta}} \times 10^2$.

$R_D(\eta)$ reproducibility of η, *i.e.*, the difference of samples of two values of η, one obtained by one operator using one set of apparatus and the other obtained by the same or a different operator using a different set of apparatus.

$r_D(\eta)$ coefficient of reproducibility of $\eta = \dfrac{R_D(\eta)}{\bar{\eta}} \times 10^2$.

$\rho(\eta)$ ratio of reproducibility of η to repeatability of $\eta = \dfrac{r_D(\eta)}{r_r(\eta)}$.

$\overline{R}_D(\eta)$ mean reproducibility of η, *i.e.*, the difference of the mean of samples of two obtained by two different operators using two different sets of apparatus.

$\bar{r}_D(\eta)$ coefficient of mean reproducibility of $\eta = \dfrac{\overline{R}_D(\eta)}{\bar{\eta}} \times 10^2$.

Discussion of Results

6.1. *Introduction*

In this section the significance of the results obtained by statistical analysis is discussed in detail. In Section 6.2 the range and accuracy of determination of the times of flow are considered. Section 6.3 contains a discussion of the values of the calibration constant A as determined for the IP Master viscometers. Section 6.4 summarizes the information on the relative accuracy associated with the methods of filling and using the IP Master viscometers. Mean values of the calibration constant A for the viscometers Nos. 0 to 4 are given in Section 6.5, and mean values of the coefficient of variation of the calibration constant K for the various viscometers are summarized in Section 6.6; a distinction is made in Section 6.5 and 6·6 between the U-tube and the suspended-level types of viscometers. In Section 6.7 the significance of the values derived for the repeatability and reproducibility of the kinematic viscosities of the standard oils in the viscometers Nos. 0 to 4 is considered.

6.2. *Range and Accuracy of Times of Flow*

In calibration work on viscometers it is recommended in "Standard Methods" * that the times of flow should not be less than 200 sec, and in ordinary routine work the lower limit recommended is 120 sec. Table 6.1 shows the frequency of the mean times of flow lying within various limits for all viscometers which have been used in the present investigation.

* "Standard Methods for Testing Petroleum and its Products," 10th edition, 1949, Institute of Petroleum.

From this table it will be seen that for all the viscometers, 10 per cent of the mean times of flow are less than 120 sec and 33 per cent are less than 200 sec. For the IP Master viscometers, 20 per cent of the mean times of flow are less than 120 sec and 59 per cent are less than 200 sec, while for Nos. 0 to 4 viscometers, 1 per cent of the mean times of flow are less than 120 sec and 10 per cent are less than 200 sec.

In "Standard Methods" it is prescribed that determinations shall be repeated until two successive times of flow agree to within 0·2 per cent.

TABLE 6.1

Frequency of Mean Times of Flow in Different Ranges

Viscometer no.	$\bar{T}<160$	$100<\bar{T}120$	$120<\bar{T}<200$	$\bar{T}>200$	Totals
IP Master .	8	30	75	77	190
0 and 1 . .	2	0	3	58	63
1A . .	0	0	3	8	11
2 . . .	0	0	5	52	57
3 . . .	0	0	6	50	56
4 . . .	1	0	2	26	29
Totals .	11	30	94	271	406

TABLE 6.2

Frequency of Coefficient of Variation of Individual Times of Flow in Different Ranges

Viscometer no.	$v(T)$ <0·0625	0·0625 <$v(T)$ <0·08	0·08 <$v(T)$ <0·10	0·10 <$v(T)$ <0·20	0·20 <$v(T)$ <0·45	Totals
IP Master .	141	22	15	12	0	190
0 and 1 .	44	12	3	4	0	63
1A . .	9	2	0	0	0	11
2 . .	33	13	3	6	2	57
3 . .	25	12	7	9	3	56
4 . .	1	11	5	9	3	29
Totals .	253	72	33	40	8	406

This condition presumably applies to runs within the same filling. In the present analysis this condition has been adhered to for the times of flow in successive runs of the same filling, but times of flow for different filling often differ by more than 0·2 per cent.

It is interesting to consider the effect of assuming that the times of flow in runs of the same or different fillings should lie within the 0·2 per cent range. Assuming that this is the desirable coefficient of repeatability in the times of flow, it follows that the corresponding coefficient of variation of the times of flow for ten observations is $v(T) = 0{\cdot}0625$, with nine degrees of freedom.

Table 6.2 gives the frequency of occurrence of values of $v(T)$ within various limits for all viscometers. From this table it is seen that the relative accuracy of the times of flow in the IP Master and Nos. 0 to 2 viscometers is generally better than that in the viscometers Nos. 3 to 4. It may be noted that of the eight entries in the range $0{\cdot}20<v(T)<0{\cdot}45$ six come from laboratory 4*a* and the remaining two from laboratory 10.

The suggestion is made that if the coefficient of variation of a series of about ten times of flow is greater than 0·20, the values are strongly suspect,

TABLE 6.3

Mean Values of Coefficient of Variation of Individual Times of Flow and Total Number of Observations

Type of viscometer	Liquid	Mean $v(T)$	Total n	Laboratories omitted
IP Master :				
U-tube Fenske filling	Water	0·051	150	—
,, upper bulb	,,	0·056	205	—
,, lower bulb	,,	0·059	203	—
,, double bulb	,,	0·032	209	Lab. 10 (n = 16)
Suspended level	,,	0·069	89	—
U-tube Fenske filling	Benzene	0·058	150	—
,, upper bulb	,,	0·069	185	—
,, lower bulb	,,	0·072	187	—
,, double bulb	,,	0·052	210	—
Suspended level	,,	0·065	90	—
U-tube double bulb and suspended level	Low cetane secondary standard	0·043	166	—
Nos. 0 and 1	,,	0·054	167	—
,, ,,	High cetane secondary standard	0·061	156	—
,, ,,	Various oils	0·064	192	—
No. 1A	,,	0·054	62	—
,,	,,	0·043	50	—
No. 2	,,	0·058	184	Lab. 4a (n = 26)
,,	,,	0·068	225	Lab. 4a (n = 20)
,,	No. 1	0·048	98 *	—
No. 3	Various oils	0·089	234	Lab. 4a (n = 20)
,,	,,	0·089	197	—
,,	PD 542	0·054	80	Lab. 10 (n = 10)
No. 4	Various oils	0·096	181	Lab. 4a (n = 22)
,,	PD 543	0·102	68	Lab. 10 (n = 12)
	Total		3738	124

* Including 20 values in Viscometer No. 1A.

and the series should be repeated at least twice for confirmation or rejection.

For convenience the mean values of $v(T)$ and the total number of observations n, for all viscometers are summarized in Table 6.3.

6.3. *Values of* A *for IP Master Viscometers*

The values of the calibration constant A for the IP Master U-tube and IP Master suspended-level viscometers are given in Table 2.3, and the mean values of A in Table 2.4. The values of A are relatively small. The effect of A on the calculated kinematic viscosity of benzene occurs in the fourth significant figure, and for most purposes A can be omitted.

If the kinematic viscosity of water is assumed to be exactly $\overline{\eta} = 1{\cdot}0068$ at 68° F, and the values of A are taken as zero, the following mean values of $\overline{\eta}$ and of the standard deviation $s(\overline{\eta})$ of the mean, for the kinematic viscosity of water at 100° F and 130° F are found from the twenty-nine available determinations.

Temp., ° F	$\bar{\eta}$	$s(\bar{\eta}) \times 10^4$	$2s(\bar{\eta})$ range	Value used
100	0·68886	0·65	0·6890–0·6887	0·6891
130	0·51848	0·80	0·5186–0·5183	0·5183

These values are only slightly different from those used in the statistical analysis.

6.4. *Relative Accuracy of Methods of Filling and Using the IP Master Viscometers.*

The relative accuracy of the kinematic viscosity of benzene as determined in the five methods of filling the IP Master viscometers, has been discussed in Section 2.5. The double-bulb method gives more accurate values than those for the other four methods. This result can be ascribed to the fact that the times of flow with the double-bulb method are generally over 200 sec, while the times of flow using the other methods are generally under 200 sec. The values of the coefficient of variation of the individual times of flow in the double-bulb method are also significantly less than the values for the other methods.

6.5. *Values of* A *for Nos.* 0 *to* 4 *Viscometers*

The individual values of A for the viscometers Nos. 0 to 4 are given in Tables 3.4, 3.8, 4.12, and 4.16. The mean values of A and the number of

TABLE 6.4

Nos. 0 *to* 4 *Viscometers. Values of* $\bar{A}$ *and Number of Observations* m

Type of viscometer	A	m	A "St. Methods"
No. 1 Suspended level	1·3	3	—
Nos. 0 and 1 U-tube	1·1 *	7	2·4
No. 1A Suspended level	2·2	3	2·8
No. 2 Suspended level	1·1	3	2·8
No. 2 U-tube (including some No. 1) . .	1·1	5	2·4
No. 3 Suspended level	22	3	2·8
No. 3 U-tube (including some No. 2) . .	27	7	7·0
No. 4 Suspended level	210	2	2·8
No. 4 U-tube (including some No. 3) . .	−9	5	11·4

* Laboratory 9*b* omitted.

observations m, classified for the U-tube and suspended-level types, are shown in Table 6.4. A column is also added indicating the values of A given in "Standard Methods."

An inspection of the table shows that the two sets of values of A do not agree, but in view of the small number of observations and the large errors which may arise in determining A in any particular case, no strong conclusion can be drawn from the data available. It seems reasonable to state, however, that for accurate work the values of A should be determined experimentally for each viscometer.

6.6. *Accuracy of Calibration*

When a viscometer is calibrated by using the mean times of flow of two oils at the same or different temperatures, or one oil at two temperatures,

the values of K and A are given by eqns (3.7) and (3.8) in the form

$$K = \frac{\bar{\eta}_1\overline{T}_1 - \bar{\eta}_2\overline{T}_2}{\overline{T}_1^2 - \overline{T}_2^2} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (6.1)$$

$$A = \frac{\bar{\eta}_1\overline{T}_1\overline{T}_2^2 - \bar{\eta}_2\overline{T}_2\overline{T}_1^2}{\overline{T}_1^2 - \overline{T}_2^2} \quad . \quad . \quad . \quad . \quad . \quad . \quad (6.2)$$

In order to obtain accurate values of K and A from eqns (6.1) and (6.2), the values of $\overline{T}_1$ and $\overline{T}_2$ and hence $\bar{\eta}_1$ and $\bar{\eta}_2$ must not be too nearly equal. If the values of $\overline{T}_1$ and $\overline{T}_2$ are nearly equal, significant figures are lost in evaluating the quotients, which in the extreme case of equal times become indeterminate.

This principle is illustrated for viscometer No. 3 in Table 4.12 in the calibration for laboratory 7. Times of flow for five calibrating oils were available. The values of K and A for the pairs of oils with times of flow close together are seen to be considerably different from those for the pairs of oils with times of flow sufficiently far apart.

TABLE 6.5

All Viscometers. Mean Values of $\nu(K)$

U-tube viscometers	Mean $\nu(K)$	m	Suspended level viscometers	Mean $\nu(K)$	m
IP Master :			IP Master .	0·012	3
U-tube Fenske filling .	0·009	5			
„ upper bulb . .	0·011	7			
„ lower bulb . .	0·011	7			
„ double bulb . .	0·006	7			
Nos. 0 and 1 . . .	0·022	8	No. 1 . .	0·022	3
			No. 1A . .	0·020	3
No. 2 (including some No. 1)	0·030	6	No. 2 . .	0·019	3
No. 3 (including some No. 2)	0·042	7	No. 3 . .	0·021	3
No. 4 (including some No. 3)	0·048	5	No. 4 . .	0·039	3

This general principle requires further investigation with the object of devising a statistical test of significance of the difference between the calculated values of K or A under the varying circumstances. No hard-and-fast rule can be laid down, and each case should be treated on its merits, but a reasonable assumption is that $\overline{T}_1$ should be at least 50 per cent greater than $\overline{T}_2$.

Further, if the mean time of flow $\overline{T}_1$ is considerably greater than $\overline{T}_2$, the calculated value of K depends essentially only on $\overline{T}_1$ and $\bar{\eta}_1$, and the parameters for the second oil contribute little to the evaluation of K. Under these circumstances the calculated value of A is subject to large errors.

This principle is illustrated for viscometer No. 4 in Table 4.15 in the calibration for laboratory 1. The mean times of flow and kinematic viscosities are as follows :—

Oil	Temp., °F	$\overline{T}$	$\bar{\eta}$	$\frac{\bar{\eta}}{\overline{T}}$
7	68	1447·12	14020	0·96882
7	130	67·36	650·83	0·96620

TABLE 6.6

All Viscometers. Values of Coefficients of Repeatability, Reproducibility, Mean Reproducibility, and Numbers of Degrees of Freedom

Temp., °F	Oil	Viscometer	$r_T(\eta)$	N	$r_D(\eta)$	N	$\rho(\eta)$	$\bar{r}_D(\eta)$
		(a) *IP Master*						
68	Benzene	U-tube Fenske filling	0·188	45	0·308	49	1·6	0·278
68	,,	,, upper bulb	0·191	58	0·254	64	1·3	0·214
68	,,	,, lower bulb	0·178	60	0·368	66	2·0	0·346
68	,,	,, double bulb	0·115	63	0·182	69	1·6	0·163
68	,,	Suspended level	0·148	27	0·372	29	2·5	0·358
100	,,	U-tube Fenske filling	0·154	45	0·224	49	1·45	0·196
100	,,	,, upper bulb	0·228	54	0·309	59	1·35	0·264
100	,,	,, lower bulb	0·238	54	0·326	59	1·4	0·280
100	,,	,, double bulb	0·177	63	0·230	69	1·3	0·194
100	,,	Suspended level	0·184	27	0·208	29	1·1	0·160
130	,,	U-tube Fenske filling	0·166	45	0·400	49	2·4	0·382
130	,,	,, upper bulb	0·194	54	0·344	59	1·8	0·312
130	,,	,, lower bulb	0·204	54	0·357	59	1·75	0·325
130	,,	,, double bulb	0·146	63	0·204	69	1·4	0·175
130	,,	Suspended level	0·232	27	0·238	29	1·0	0·173
68	Low cetane	U-tube double bulb	0·110	75	0·212	83	1·9	0·199
130	,,	Suspended level	(0·116) 0·134	(72) 77	(0·306) 0·415	(79) 85	(2·6) 3·1	(0·299) 0·403
		(b) *IP Standards*						
68	High cetane	Nos. 0 and 1	(0·142) 0·219	(42) 68	(0·455) 0·805	(49) 78	(3·2) 3·7	(0·446) 0·793
130	,,	,,	(0·139) 0·177	(43) 66	(0·358) 0·882	(50) 76	(2·6) 5·0	(0·341) 0·878
68	No. 1	Nos. 1A and 2	(0·154) 0·156	(70) 88	(0·382) 0·551	(77) 97	(2·5) 3·5	(0·367) 0·540
68	PD 542	No. 3	(0·183) 0·295	(62) 81	(0·398) 0·836	(69) 89	(2·2) 2·8	(0·377) 0·810
68	PD 543	No. 4	(0·312) 0·577	(61) 72	(1·40) 1·38	(67) 79	(4·5) 2·4	(1·38) 1·31

TABLE 6.7

Mean Values

Viscometer	$r_T(\eta)$	N	$r_D(\eta)$	N	$\rho(\eta)$	$r_D(\eta)$
IP Master (double bulb) . .	(0·135) 0·139	(336) 341	(0·231) 0·263	(369) 375	(1·7) 1·9	(0·212) 0·244
Nos. 0 and 1	(0·140) 0·199	(85) 134	(0·409) 0·844	(99) 154	(2·9) 4·2	(0·396) 0·837
Nos. 1A and 2	(0·154) 0·156	(70) 88	(0·382) 0·551	(77) 97	(2·5) 3·5	(0·367) 0·540
No. 3	(0·183) 0·295	(63) 81	(0·398) 0·836	(69) 89	(2·2) 2·8	(0·377) 0·810
No. 4	(0·312) 0·577	(61) 72	(1·40) 1·38	(67) 79	(4·5) 2·4	(1·38) 1·31

Figures in brackets denote values when abnormal results from certain laboratories are omitted.

The value of K found by the formula (6.1) using the times of flow and kinematic viscosities of the two oils is $K = 0{\cdot}96883$, so that the contribution of a second oil only effects the last figure by a unit, even though the two values $\frac{\bar{\eta}}{\bar{T}}$ are considerably different.

The mean values of the coefficient of variation of K and the number of observations for the viscometers Nos. 0 to 4, divided into the two categories of U-tube and suspended level are shown in Table 6.5. Owing to the small number of observations, no definite conclusions can be drawn about the relative accuracies of the U-tube and suspended level types, although there is a tendency for the suspended level viscometers Nos. 2 to 4 to appear to be more accurate than the U-type viscometers Nos. 2 to 4.

6.7. *Values of Repeatability and Reproducibility*

The calculated values of the coefficients of repeatability, reproducibility, and the number of degrees of freedom for the various standards are collected together in Table 6.6. In Table 6.7 the information in Table 6.6 is condensed to show the mean values of the repeatability, reproducibility, and mean reproducibility. The figures shown in brackets in both tables denote the values obtained when abnormal results from certain laboratories are omitted.

The results indicate that abnormal values from 1 or 2 laboratories tend to dominate the composite figures. Considering the values for all the laboratories, it is seen that the values for viscometers No. 1 do not fall into

TABLE 6.8

Estimated Values of Repeatability and Reproducibility

Order of magnitude of viscosity, cs	No. of steps in calibration	$r_r(\eta)$	$r_R(\eta)$	$\rho(\eta)$
1	1 [1]	0·14	0·25	1·8
10	2 [2]	0·16	0·4	2·5
100	3 [3]	0·2	0·5	2·5
500	4 [4]	0·3	0·7	2·3
1500	5 [5]	0·5	1·5	3

[1] *E.g.*, IP Master.
[2] *E.g.*, IP Master + No. 0 or IP Master + No. 1.
[3] *E.g.*, IP Master + No. 0 + No. 1 or IP Master + No. 1 + No. 1A or IP Master + No. 1 + No. 2.
[4] *E.g.*, IP Master + No. 0 + No. 1 + No. 2 or IP Master + No. 1 + No. 1A + No. 2 or IP Master + No. 1 + No. 2 + No. 3.
[5] *E.g.*, IP Master + No. 0 + No. 1 + No. 2 + No. 3 or IP Master + No. 1 + No. 1A + No. 2 + No. 3 or IP Master + No. 1 + No. 2 + No. 3 + No. 4.

the general pattern, since they are relatively too large. The values of the parameters for oil PD 543 in viscometers No. 4 are also unexpectedly high.

Table 6.8 gives tentative estimates of the values of the repeatability and reproducibility for the kinematic viscosities of oils in all viscometers which might be adopted pending more experimental information.

SOME EXPERIMENTS ON THE INFLUENCE OF A SOLID PHASE ON THE OXIDATION OF LOW VISCOSITY MINERAL OIL

By D. J. W. Kreulen (Fellow)

Summary

The presence of a solid phase affects both the length of the induction period and the rate of oxidation of a mineral oil. Besides glass, other solids such as sand, anthracite, retort carbon, and SiC are effective. There is no correlation between length of induction period and rate of oxidation during the following reaction period. Experiments carried out with glass, quartz, and sand point to a decreasing catalytic effect of the solid phase when its chemical resistance increases. The nature of the phenomenon is still obscure and needs further investigation.

In previous papers [1] it has been shown that the presence of glass increases the oxidation velocity of mineral oils. Some experiments have now been carried out using different types of glass and other materials such as anthracite and carborundum.

The oil used in the experiments was a transformer oil: viscosity 20° C, 33 cs; molecular weight (cryoscopic), 330; chemical constitution according to n-d-M method,[2]—aromatic rings, 2 per cent; naphthenic rings, 35 per cent; paraffinic side chains, 63 per cent; rings per molecule, 2·4.

The oxidation was performed at 140° C with oxygen in the laboratory's standard apparatus [3] at a stirring velocity of 1400 r.p.m. The material to be investigated was ground to between 65 and 200 mesh (Tyler) and added in quantities such that 4 g of solid per 100 cc of oil always was present. The application of equal surface areas instead of weights would have been preferable, and it should be noted that the specific gravities of the various materials were as follows: sands and glasses, approx 2·5; quartz, 2·1; retort carbon and anthracite, 1·4; carborundum, 3·1.

During the experiment the solid was renewed every 24 hr during the induction period by settling and introducing a new portion and every 2 hr during the reaction period. In most experiments after 12 hr of actual reaction no fresh solid was added, but the experiment continued without the solid phase so as to investigate whether the reaction slowed down.

In Fig 1 the unbroken lines refer to the part of the experiment in which a solid phase was present. When the solid phase was removed the unbroken line changes into a dotted line.

Experiments have been carried out with the oil in its normal state (0) and also in the presence of different materials as listed in Table I.

The water solubility of alkali from glasses 2, 3, and 4 was in the proportion 20, 7, and 1. Because of the results obtained with these three glass types the effect of small quantities of KOH was investigated, but did not increase the oxidation velocity of the oil.

The oxidation of the oil was followed by measuring its acid value by the IP 1/46 but using as small a quantity of oil as possible. The de-

crease in volume resulting from the withdrawal of samples from the reaction vessel was taken into account when fresh portions of solid were added.

The results obtained are reported in Table I and Fig 1. When no solid phase is present the increase in acid value of the oil is so slow that it is only experiments extending over very long periods of time that show a definite reaction. When a solid phase is present the rate of reaction increases suddenly after a certain time (induction period), and the next period (reaction period) is characterized by a fairly quick increase in acid value.*

During the reaction period a brownish compound is deposited on the surface of the solid phase and on the wall of the reaction vessel. This

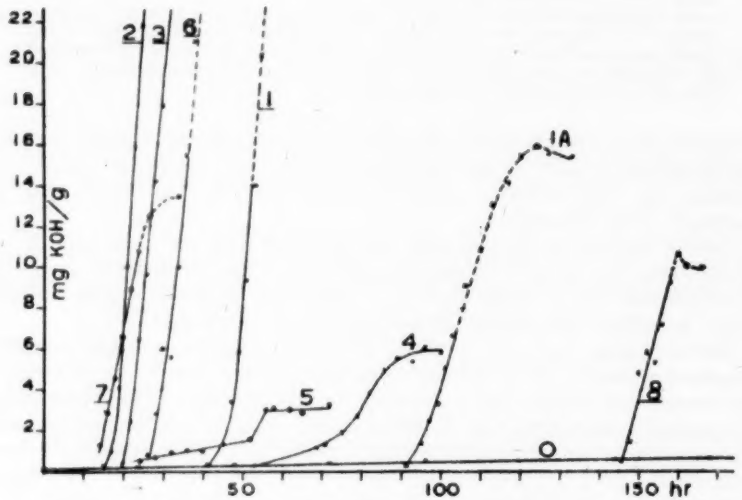


FIG 1

compound is soluble in hot alcohol and apparently identical to the " resins " formerly investigated.[1] Resins also precipitate from the oxidized oil during storage, increasingly so as the oil is further oxidized.

When the solid is removed during the beginning of the reaction period the reaction stops rather suddenly. When, however, the oil is in a more advanced stage of oxidation the reaction will generally proceed after removal of the solid.

From Fig 1 it follows that the presence of a solid phase affects both the length of the induction period and the rate of oxidation of the oil during the reaction period. Besides glass, other solids such as sand, anthracite, retort carbon, and SiC are also effective. There is no correlation between length

* The experiments with and without solid phase also differ with respect to the change in colour of the reacting oil. When no solid phase is present it changes quickly to dark brown and even black, while when a solid phase is present this change proceeds very slowly and never reaches the intensity previously mentioned.

TABLE I

Exp. 0		Exp. 1		Exp. 1A		Exp. 2		Exp. 3		Exp. 4		Exp. 5		Exp. 6		Exp. 7		Exp. 8	
hr	Acid value	hr	Acid value	hr	Acid value	hr	Acid value	hr	Acid value	hr	Acid value	hr	Acid value	hr	Acid value	hr	Acid value	hr	Acid value
0	0·1	41	0·3	91	0·3	15	0·3	20	0·4	53	0·3	24	0·5	26	0·8	14	1·3	146	0·5
24	0·2	45	1·3	95	1·4	17	1·0	22	2·4	69	1·2	28	0·7	28	2·8	16	2·9	148	1·5
48	0·3	47	3·4	97	2·5	19	4·1	24	6·4	71	1·3	32	0·9	30	6·0	18	4·6	150	4·8
72	0·4	49	5·8	99	3·3	21	10·0	26	9·6	75	1·9	36	1·0	32	5·6	20	6·6	152	5·8
96	0·5	51	9·3	101	5·1	23	15·8	28	14·2	79	2·7	40	1·0	34	10·0	22	8·9	154	5·4
120	0·5	53	14·0	103	6·7	25	21·8	30	17·9	82	3·8	52	1·6	36	15·5	24	10·8	156	7·2
144	0·6	55	20·4	106	9·1	27	29·2	32	23·4	86	5·0	56	3·0	38	21·1	27	12·5	158	9·3
168	0·7	57	25·4	110	10·9	29	35·5	35	27·0	89	5·6	58	3·1	40	23·7	34	13·5	160	10·7
192	0·8	59	29·7	113	13·1	31	41·1	37	28·3	93	5·4	62	3·0	44	34·4			162	10·1
216	0·9	61	34·4	117	14·2	33	46·7	39	31·9	96	6·1	65	2·9	47	35·1			166	10·0
240	1·0	63	36·0	120	15·5	36	52·6	42	33·8	100	5·8	72	3·3	51	41·1				
		66	40·8	124	16·0	38	57·3	44	34·4										
		70	44·0	127	15·6	40	58·2	47	38·7										
		73	55·6	133	15·5	43	62·0	50	40·7										
		80	56·5			45	65·4												
		83	60·4			48	65·1												
						51	69·9												

Exp. 0. Blank experiment.
„ 1. Sand added, 1·6% Fe.
„ 1A. „ „ HCl treated sand, 0·4% Fe.
„ 2. White glass, approx 72% SiO_2, 10% CaO, 16% Na_2O.
„ 3. Green glass, approx 65% SiO_2, 6% Al_2O_3, 2% Fe_2O_3 + Mn_3O_4, 13% CaO and 14% Na_2O.
„ 4. Laboratory glass, approx 75% SiO_2, 5% Al_2O_3, 8% B_2O_3, 4% ZnO, 2% CaO, 6% Na_2O.
„ 5. Broken laboratory quartz.
„ 6. Retort carbon, ash 2·4%.
„ 7. Anthracite, volatile matter 9%, ash 1·8%.
„ 8. Carborundum (SiC).

Horizontal line in column indicates removal of solid phase.

of induction period and rate of oxidation during the reaction period. The experiments with glass and quartz, and also those with sand, suggest a decreasing catalytic effect of the solid phase when its chemical resistance increases. The nature of the phenomenon, however, is still obscure, and needs further investigation.

Rotterdam, Laboratory for Oil and Coal Chemistry.

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ON THE INFLUENCE OF METAL, ESPECIALLY COPPER, ON THE OXIDATION OF WHITE OIL

By D. J. W. Kreulen (Fellow)

Summary

Previous work carried out by the author on the oxidation of white oil is reviewed, and the factors necessary for a reliable oxidation test are formulated.

When white oil is treated with oxygen at 100° C oxidation does not start immediately. During the induction period, no reaction or only a very slight one, takes place; this is followed by a period of actual reaction. The effect of copper on the length of the induction period and on the reaction velocity during the reaction period is discussed.

When copper is present the length of the induction period is shorter than when copper is absent, the more so when more copper is added. Finally, the length of the induction period becomes zero. The curve which shows the relationship between the length of the induction period and the quantity of copper which has been added suggests that an adsorption phenomenon is being dealt with. It is reasonable to assume that anti-oxidants are adsorbed on the copper surface. This assumption is supported by the fact that tin, which has a lower relative affinity of adsorption than copper, is less active in shortening the length of the induction period than copper. When very large quantities of copper are added an induction period reappears. This might indicate the breaking of reaction chains.

As to the effect of copper on the reaction velocity in the reaction period; up to about 500 cm^2 of copper surface added to 250 ml of oil do not affect the reaction velocity appreciably. From experiments carried out with larger quantities of copper, however, it is evident that copper still has a catalytic effect. It is shown that this catalytic effect is due in part to the solid copper and in part to the copper that went into solution.

Experiments carried out with copper as such, copper reduced in a stream of hydrogen, and also with copper oxide are discussed. The activity of Cu_2O and Cu is found to be of about the same order.

A method is given for the determination of slight amounts of dissolved copper in oil.

In the period between 1940 and 1943 the author and his co-workers carried out work on the influence of metals, especially copper, on the oxidation of white oil. Results of this work were published during the war,[1] and the whole investigation is summarized here. More recently other aspects of the subject have been discussed.[2]

Many investigations on the oxidation of mineral oils have been carried out with transformer oils. This is understandable, as oxidation stability is essential, but transformer oils contain naphthenic rings and paraffinic side chains and also aromatic rings which influence the oxidation stability of the molecule considerably. Therefore, for simplification investigations were carried out with white oils which are more simple in structure owing to the almost total absence of aromatic rings. Thus, results will be more readily interpretable; but on the other hand, the conclusions which are drawn have to be applied with reserve to other oils, especially to those containing aromatic rings.

When a white oil is treated at 100° C with air or oxygen, oxidation starts only after an appreciable period of time, the induction period. In Fig 1 this is shown for two oils, oil No. 1 having a longer induction period than

oil No. 2, but when the induction period has ended its reactivity is higher because angle α_1 is greater than α_2.

In this example, where the complete course of both induction period and reaction period can be studied, results are easy to estimate. But there are many tests in which the oil is assessed according to its state of oxidation after only one fixed period of time. That this procedure might be unreliable emerges from further study of Fig 1. For example, if an estimate had been made of the oils after a fixed period of time t, the conclusion would have been that oil 2 was more reactive than oil 1.

Consequently, it is necessary to estimate the oxidation stability of an oil by both length of induction period and reaction velocity during the reaction period.

To determine the length of the induction period the constant, by which the oxidation of the oil is followed, must be carefully selected. The acid value, for instance, is unsuitable for this purpose, as in the case of white oils the relationship between acid value and oxidation time is a strongly

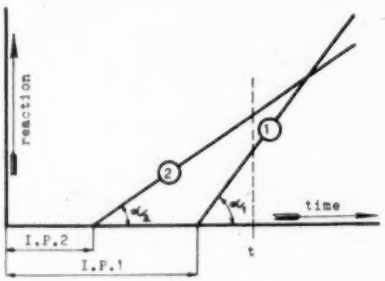

FIG 1

curved one. Consequently, it is not possible to conclude with certainty when the induction period ended.

What is needed is a constant which shows a straight-line relationship to time so that a graph as shown in Fig 1 is obtained. In that case the point of intersection of the straight line of reaction and the time axis may be accepted as the end of the induction period.

The dipole moment, which fulfils this requirement, was employed in the investigations, and is preferred because when an oxygen molecule attaches itself to an oil molecule the first result will be the introduction of a polar group into the system. Thus it is to be expected that the dipole moment will indicate immediately any change in the oil.

A general remark must be made in connexion with oxidation tests, in most of which the gaseous phase is dissolved into the oil, either by stirring or by bubbling. During the test the dissolved gas is consumed by the reaction. It is therefore essential that if true reaction velocities are to be measured the dissolving velocity be greater than the consumption velocity, because oxygen consumption is limited by oxygen supply, and even the most reactive oil will oxidize only slowly when the reacting gas is supplied at a low rate.

Many experiments have been carried out by the author and his co-

workers [1] to establish conditions which guarantee a sufficient supply of oxygen. It has been found that these conditions are rather severe. Stirring velocities above 1000 r.p.m. and a specially devised apparatus [3] have to be used.

This implies that in routine tests the *conditio sine qua non* for the measurement of reaction velocities : dissolving velocity is greater than consumption velocity, will seldom be fulfilled.

Irving and Thompson [2] describe experiments in which the access of oxygen has been consciously limited. From the analysis of the oxidized oil it appeared that in that case the product proportions are significantly different from those obtained in experiments with an excess of oxygen.

Because many routine tests provide information about the oil's behaviour in common practice and because in practice the access of oxygen is often limited by constructional features, experiments with limited supply of oxygen are certainly not as unacceptable as might be thought on first consideration. Then, however, because this type of test is extremely sensitive to small variations in the supply of oxygen, special precautions must be taken to ensure an absolutely constant supply of oxygen.

Consequently, the criteria for reliable oxidation tests are :—

(*a*) Where real reaction velocities are wanted dissolving velocity of oxidizing agent should be greater than consumption velocity;

(*b*) Where it is desired to imitate certain conditions dissolving velocity of oxidizing agent may be lower than consumption velocity but closely controlled;

(*c*) in both cases measurement of the length of the induction period and the rate of reaction in the reaction period separately.

In the experiments quoted below the practices mentioned under (*a*) and (*c*) were followed. Apparatus and experimental technique has been previously described.[4] The white oil used had the following constants : mol. wt. (cryoscopic), 450; chemical constitution according to n-d-M method, naphthenic rings 37 per cent, paraffinic side chains 63 per cent average number of rings per molecule 2·8.

Varying quantities of filings of electrolytic copper were used as a catalyst. The quantity of copper added is indicated in the graphs as g/250 ml of oil. The filings passed a 400-mesh sieve. From their specific weight, together with a microscopic determination of their particle dimensions, it followed that 1 g of copper filings represented 45 cm^2 of surface.

The experiments were carried out at 100° C. At regular intervals they were interrupted and the dipole moment $\bar{\mu}$ of the oil was measured.

This dipole moment was estimated as the difference between the molecular polarization P and the molecular refraction Po at a temperature of 20° C.

$$\bar{\mu} = 0{\cdot}0127 \,.\, 10^{-18} \sqrt{(P - Po)T}$$

$$P = \frac{\varepsilon - 1}{\varepsilon + 2} \cdot \frac{M}{d} \qquad Po = \frac{N^2 - 1}{N^2 + 2} \cdot \frac{M}{d}$$

where ε = dielectric constant; M = molecular weight; d = density, N = refractive index; $\bar{\mu}$ = mean dipole moment; T = absolute temperature.

The dielectric constant was measured by a high-frequency method.

I. *The Length of the Induction Period*

The relationship between the amount of copper added and the length of the induction period is shown in Fig 2. The curve consists of two parts, *A* and *B*. In the beginning, when increasing amounts of copper are added, the induction period decreases sharply in length. Fairly soon it becomes zero, which means that when, for example, 20 or 30 g of copper

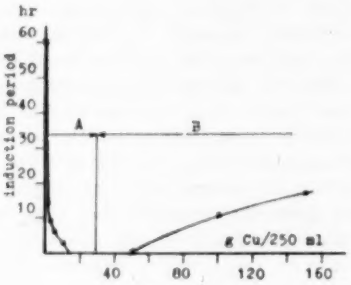

FIG 2

filings are added the reaction starts immediately. When, however, very large amounts of copper are added (50 g and more) an induction period reappears.

In Fig 3 part *A* of Fig 2 is plotted on a larger scale. The shape of the curve suggests that adsorption is taking place. In order to verify this, experiments have been made with $2\frac{1}{2}$ g copper for different periods of time

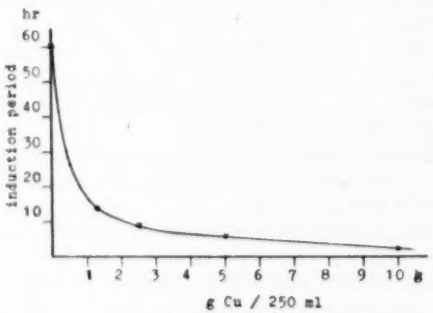

FIG 3

varying from 1 up to 9 hr. After the chosen period of time the copper was settled and removed and the experiment continued without copper in order to estimate the length of the induction period of the treated oil. The results are reported in Fig 4.

According to Moureux and Dufraisse [5] the induction period is caused by the presence of anti-oxidants which exercise their influence in minor quantities. This idea being accepted, the induction period of the samples mentioned in Fig 4 is the time necessary to destroy that part of the anti-

oxidant which has not been adsorbed to the metal surface. This explains why about the same length of induction period has been found in the experiments of Fig 4. It confirms the suggestion of Fig 3 that an adsorption phenomenon has occurred.

According to Vellinger and Saito [6] most anti-oxidants present in mineral oil contain polarizable groups. It is generally known that copper has a higher relative affinity of adsorption for polarizable compounds than tin, and therefore tin would be expected to be less active in shortening the length of the induction period than copper. This was confirmed by experiment. The induction period of the oil without any supply of metal amounted to 60 hr. By adding 1200 cm^2 of copper it was reduced to 8 hr, and by adding the same amount of tin to 36 hr.

Regarding the reappearance of an induction period when very large quantities of copper are used (part *B* of Fig 2), this phenomenon has nothing to do with the presence of anti-oxidants. It is known that solids often act as chain-breakers when they are present in reacting gases or

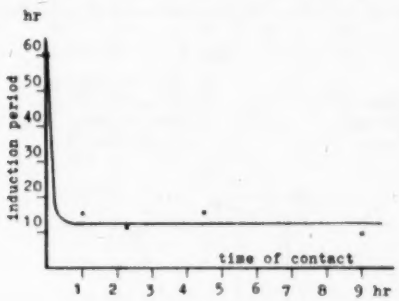

FIG 4

liquids. Therefore it seems reasonable to explain the reappearance of the induction period by a chain-breaking action of the large quantities of solid phase.

II. *The Reaction Velocity in the Reaction Period*

In the reaction period the dipole moment $\bar{\mu}$ increases linearly as a function of time, and thus the angles of slope of these lines or their tangents can be used to express the reaction velocity of the oil in the reaction period. In the following experiments the tangents were used.

In Figs 5 and 6 the results of two series of experiments are shown. Fig 5 deals with the effect of relative small quantities (0 to 10 g) of copper, while Fig 6 refers to experiments in which larger (25 to 150 g) quantities were used. It should be recalled here that 1 g of copper corresponds to about 45 cm^2 of surface.

In Fig 6 zero indicates the end of the induction period.

All the lines shown in Fig 5, slope at about the same angle. There is a small increase in angle of slope when more copper is added, but this increase is so small (tangent of angle of slope 1·2 in blank experiment and 1·4 when 10 g of copper are present) that it is very close to the experimental

error. Therefore it may be concluded that small amounts of copper up to about 500 cm^2/250 ml, have almost no effect on the reaction velocity of white oils.

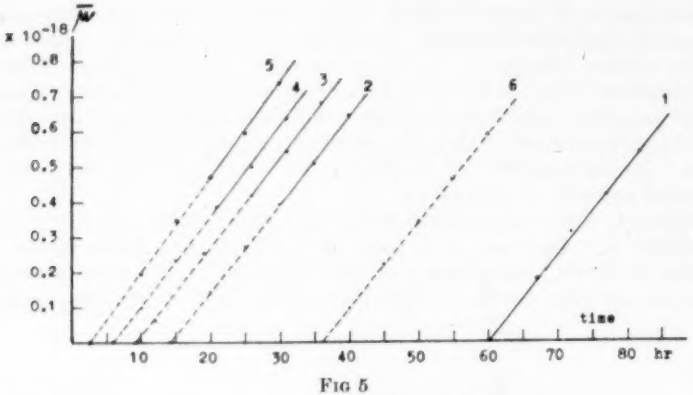

FIG 5

1. BLANK.
2. 1·25 G CU/250 ML.
3. 2·5 G CU/250 ML.
4. 5 G CU/250 ML.
5. 10 G CU/250 ML.
6. TIN ADDED. SAME SURFACE AS COPPER IN EXPT 3.

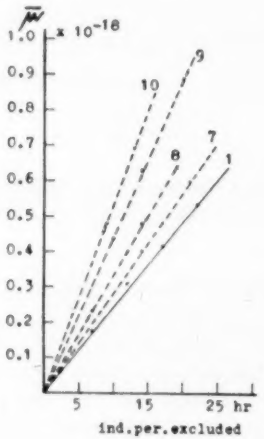

FIG 6

1. BLANK.
7. 25 G CU/250 ML.
8. 50 G CU/250 ML.
9. 100 G CU/250 ML.
10. 150 G CU/250 ML.

This conclusion is confirmed by experiments 2, 3, 4, and 5 (see Fig 5) in which the copper has been removed (indicated by the change of dotted line into unbroken line) during the run of the experiment. The lines continue their course without any change, indicating that the copper

present exerts practically no influence. In the case of large quantities of copper, however, a pronounced catalytic effect is exhibited. This is shown in Fig 6. In the experiment in which 150 g of copper are used the dipole line slopes at an angle whose tangent is 2·7.

In the experiments with large amounts of copper the oil becomes increasingly green as the reaction proceeds. It is evident that copper enters into solution, and it may be asked whether the solid copper, the dissolved copper, or both, act as a catalyst.

The experiments reported in Fig 7 provide an answer to this question.

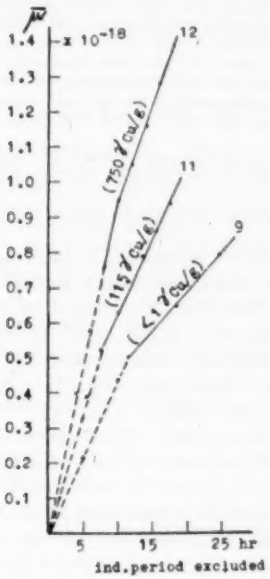

FIG 7

9. 100 G CU/250 ML.
11. 200 G CU/250 ML.
12. 300 G CU/250 ML.

To 250 ml of oil were added 100, 200, and 300 g of copper respectively. When the oil was in full reaction the copper was filtered off and the experiment continued without solid copper. In the graph the dotted lines refer to that part of the experiment in which solid copper is present. The unbroken lines refer to that part of the experiment in which the solid copper has been removed.

From the results it is clear that both the solid copper and the dissolved copper exert a catalytic action. When using 300 g of copper the line slopes at an angle the tangent of which is 4·8. When the copper has been filtered off it slopes at an angle the tangent of which is 2·8. In the blank experiment the tangent of the angle of slope is 1·2.

For the determination of the amount of copper in solution, the amount

of which is indicated in brackets (Fig 7), the following method has been developed :—

Introduce a weighed sample of the filtered oil, containing >10 γ Cu, together with 25 ml 10 per cent H_2SO_4 and a few pieces of H_2SO_4-treated Pumice-stone into a flask having a glass stopper equipped with a cooler.

Boil for ½ hr and transfer, after cooling, to a separating funnel. Allow the acid phase to settle and draw off. Rinse the flask with 10 ml distilled water, transfer this to the oil in the separating funnel, and shake well. Allow the water to settle and combine it with the acid extract. Repeat this washing. Boil the combined extracts for 5 min with 10 ml 0·01*N*-$KMnO_4$ in order to eliminate the yellow colour. Add a crystal of oxalic acid, cool, and make the solution slightly alkaline by the addition of some strong ammonia. Dilute with distilled water to exactly 100 ml and filter through hard filter-paper. Discard the first drops of filtrate.

Transfer 50 ml of the filtrate to a colorimeter tube of about 3 cm diameter which is, except for a space to allow observation of its contents, surrounded by dull black paper. Add 5 ml of an aqueous solution of 0·1 per cent Na diethylthiocarbamate and mix.

Titrate the contents of another tube containing 50 ml of distilled water and 5 ml 0·1 per cent Na diethylthiocarbamate with an aqueous solution of $CuSO_4$ (0·01 mg Cu/ml) till the colour matches that of the test solution. Observe the colour against a dull-white background.

Calculate the copper present in the test solution in the usual way.

A blank test, to correct for the Cu present in filter-paper and reagents, generally indicates about 5 γ Cu.

The method allows the estimation of Cu to within 0·5 γ/g of oil.

It has often been disputed whether Cu or Cu_2O is responsible for the shortening of the length of the induction period and for the increase in reaction velocity during the reaction period of mineral oils. Therefore, simultaneous experiments were made with ordinary copper filings, and with the copper filings carefully reduced and cooled in an atmosphere of hydrogen before putting them into the oil. In addition, experiments with copper oxide have been made. It was found that ordinary copper filings, reduced copper filings, and copper oxide also decrease the length of the induction period and increase the rate of oxidation during the reaction period. The activity of Cu_2O and Cu was found to be about of the same order. The objection might be raised that when reduced copper was used in experiments it would immediately oxidize during the experiment. This objection is valid, but it cannot explain the results of the following two experiments : 250 ml of oil were treated for 10 hr at 100° C with 300, and with 40, g of reduced copper in an atmosphere of nitrogen. The copper was then filtered off and the oil tested on its induction period in the presence of oxygen. The length of the induction period amounted to 60 hr in the blank test, but treatment of the oil with reduced copper in an atmosphere of nitrogen had reduced it to 6½ and 7½ hr respectively. This points to solid copper being itself effective, a statement which is contradictory to Krieger's conclusion [7] that Cu, free from Cu_2O, has no catalytic effect. But it must be remembered that Krieger investigated transformer oil, while the present investigation deals with white oil.

In connexion with this point may also be quoted Matthews' [8] work on the corrosion potentiality of lubricating oil towards cadmium–nickel bearing metal. Matthews also used copper as a catalyst, and there is strong evidence that the demand/dissolving velocity of oxidizing agent is greater than consumption velocity, is fulfilled in his experiments. Matthews measured both the length of the induction period and the reaction velocity during the reaction period. It is striking the extent to which

Matthews' graphs resemble Fig 5 of the present paper. The lines which show the reaction velocity during the reaction period, run practically parallel, the only significant effect is the shortening of the length of the induction period. Therefore the length of the induction period (break-down time) was accepted by Matthews as a basis for the evaluation of the corrosion potentiability of lubricating oil towards cadmium–nickel bearing metal.

In the light of Krieger's experiments, the concordance of Matthews and the author's results is the more striking. For here also different types of oil—lubricating and white—are concerned.

Rotterdam, Laboratory for Oil and Coal Chemistry.

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CLASSIFICATION OF BITUMENS IN ASPHALT TECHNOLOGY BY CERTAIN RHEOLOGICAL PROPERTIES

PART IV

CRITICISM OF CERTAIN ESTABLISHED FLUIDITY FACTORS AND DERIVATION OF MORE RATIONAL INDICES

By S. L. Neppe (Fellow)

Synopsis

Inherent errors in the Fluidity Index of Holmes, Collins, and Child are pointed out, and the method is modified to remove certain irregularities. In addition to other limitations, the Zapata Fluidity Factor is shown to be radically unsound, being affected to a far greater extent by hardness than by the quality of the bitumens.

Analytical data for numerous samples are presented and this has enabled the author to develop a rational Fluidity Characterization Factor based on a graphical study of absolute viscosity–temperature relationships over a wide range of temperatures.

A direct comparison of indices ascertained for each method clearly demonstrates the superiority of the systems recommended by the author. Evidence is provided that viscosity number, fluidity characterization factor, and the modification of the H.C.C. index are closely related, being measures of temperature susceptibility.

Experimental confirmation is also furnished of the author's previous deductions regarding viscosities of bitumens at their softening-point temperatures.

The H.C.C. Fluidity Index and its Modification by the Author

As pointed out in Part I of this series of papers,[1] the chart constructed by Holmes, Collins, and Child [4] for characterizing asphaltic bitumens by a Fluidity Index relating Saybolt Furol Viscosities at 275° F with penetration at 77° F was based on their experimental results obtained for an inferior series of petroleum tars, arbitrarily assigned index values of 0 throughout, and on a superior series of Mexican asphalts to which index values of 100 were allotted.

The original iso-penetration lines of the chart have been reproduced in Fig 30, but in this case three vertical lines *AB*, *CD*, and *EF* have been added by the author as shown.

If it is assumed that the iso-penetration lines for say 25 and 200 pen respectively are correct, the positioning of the remaining diagonal lines representing other penetrations should rationally be such as to follow some definite mathematical or physical law.

The right-hand side of line *AB* has accordingly been divided off logarithmically from 25 to 200 pen. If this scale is compared directly with that fixed by Holmes, Collins, and Child—shown on the extreme right of the chart—it is immediately evident that the divisions of the latter are certainly not logarithmic and appear to have been placed irregularly in such a way as to satisfy a limited amount of experimental data. This is

clear from the fact that, apart from the basic lines for the two fundamental penetrations of 200 and 25, only the lines for penetrations of 150 and 120 correspond fairly closely on the two scales. The degree of deviation from about 120 to 200 pen is relatively small, and by itself hardly merits criticism, but appreciable differences are apparent for penetrations below

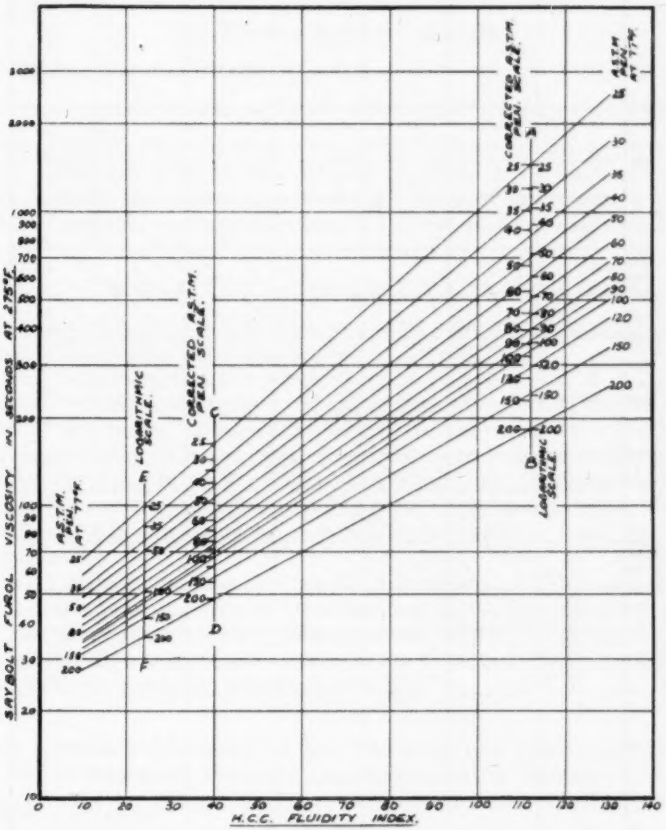

FIG 30

IRREGULARITIES IN THE H.C.C. FLUIDITY INDEX CHART

this range. Similar remarks apply to the left-hand side of the chart showing points on the line *EF*, which has also been divided logarithmically from 25 to 200 pen.

In order to produce a more rational H.C.C. Index, the author has marked off divisions on the line *CD* and on the left-hand side of *AB* to show the correct ASTM penetrations when allowance is made for the shape of the penetrometer needle. The positions of the ASTM 25 and 200 pen lines have been

left unchanged, but the intermediate points have been determined as follows :—

Reference to Table III [Part I [1]] shows that ASTM penetration values of 25 and 200 are equivalent to true penetrations of 8·8 and 177·2 respectively. Vertical lines *AB* and *CD* have accordingly been divided off logarithmically

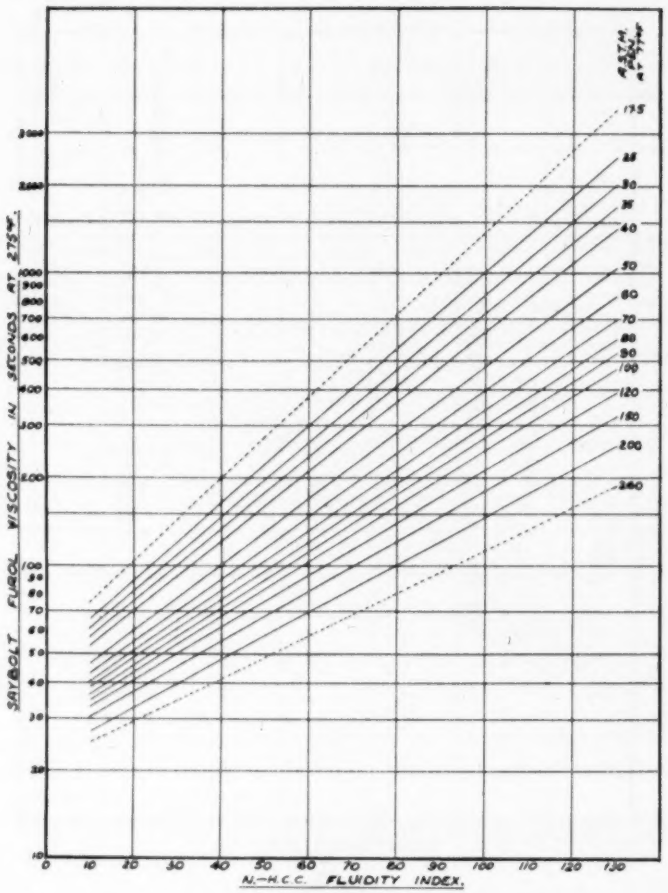

FIG 31

THE N-H.C.C. FLUIDITY INDEX CHART AS REVISED BY THE AUTHOR

from 8·8 to 177·2 to give the intermediate " true penetration " positions as discussed in Part I, from which the equivalent ASTM penetrations have easily been located.

The corresponding ASTM penetration points on *AB* and *CD* have been joined as shown in Fig 31. This diagram is the writer's modification of the H.C.C. Fluidity Index, and has been designated the N–H.C.C. Fluidity Index.

One important advantage that the author's revised method has over the original is that the chart can now be extended in either direction to give further diagonal lines for penetrations below 25 and above 200 respectively, For example, the positions corresponding to lines of 17·5 and 260 pen respectively are shown dotted in Fig 31, whereas there is no definite basis for similar extensions in Fig 30.

The Zapata Fluidity Factor

The fluidity factor of Zapata [5] is :—

[Furol viscosity in seconds at 275° F − Pen at 77° F] × Pen at 77° F/100.

Mathematically, this factor is completely irrational, since it involves a straight difference between two entirely unrelated units such as time of flow in seconds and penetration in dmm. Even if each term is altered to units of absolute viscosity in poises, the factor would be expressed in the form

Z.F.F. = − [Poises at 77° F − Poises at 275° F] × Poises at 77° F/100.

The negative sign above has no particular significance apart from adherence to the original form as proposed by Zapata.

For all bitumens, the numerical value of poises at 275° F is entirely negligible in relation to poises at 77° F, so that the effect of the former in a factor of this nature is entirely nullified. In other words, Zapata's fluidity factor converted to absolute units may in fact be reduced simply to the square of the poises at 77° F. It can thus readily be appreciated that hard bitumens in any series must inevitably have considerably higher fluidity factors than the corresponding soft products prepared from the same base material.

If the Zapata factor has any practical value at all, it can be only in direct comparisons of different bitumens having the same penetration at 77° F, so that in effect it is really the viscosities at 275° F which are actually being compared. Under such circumstances therefore, the necessity for a factor of this nature is obviously redundant.

The Author's Approach to the Problem and the Derivation of a Rational Fluidity Characterization Factor

Selection of Samples

In order to obtain a wide range of experimental data, thirty-six representative bitumen samples from various sources were selected (see Table VI). Some were obtained through suppliers of commercial bitumens, and others were specially prepared or modified in the laboratory by vacuum or steam distillation or by air-blowing, particularly in the case of the harder samples. The chief aim was to collect a range having gradually increasing Viscosity Numbers according to the author's classification [Part II [2]] and to group them, where possible, for convenience. In addition, attempts were made at procuring samples having penetrations in the region of 200, 100, and 60 respectively for each grouping of similar materials. In a few cases bitumens of lower penetration were also included. It should be emphasized, however,

that, for purposes of this phase of the investigations, it was not essential for the individual members of any one class to originate from the same parent soft substance as was originally required for the different bitumen series used in developing the various characterization indices described in Parts II [2] and III [3] of this work. In certain instances the samples from commercial sources were probably prepared in similar ways from reasonably similar base bitumens. This applies, in particular, to samples obtained from different suppliers of similar classes of products. In others the inclusion of some samples in any given grouping or class was a matter of convenience only, and was dictated by knowledge of their similarity of origin or history of preparation. Some apparent discrepancies in specific gravity and Viscosity Number noted, for example, in certain members of Groups 15A–C, 19A–C, 20A–C, and 21A–E respectively are thus readily understandable and are not really of importance for the present discussions, since all the samples were treated individually as well as, for convenience, from the angle of approximate collective groups.

Experimental Work

In view of the great importance of relating the rheological characteristics of bitumens at high and low temperatures, it was necessary to compare the susceptibilities of bitumens over the temperature range 77° to 275° F. The data of immediate interest were :—

1. ASTM penetrations at 77° F.
2. BRTA viscosities at two or three of the standard temperatures of 140°, 158°, and 176° respectively.
3. Redwood II viscosities at two or three of the following temperatures, viz., 212°, 230°, 248°, 275°, and 302° F respectively. These results were all converted to equivalent Saybolt Furol values by means of standard reference tables.
4. In addition, it was necessary to know the specific gravities at the various temperatures. Values were determined at only 60° F, but in accordance with general practice in the petroleum industry standard correction factors were used for computing the corresponding specific gravities at all other temperatures.

The experimental data obtained in a number of different units were all brought to a common basis by conversion to absolute units as follows :—

(i) For the conversion of the ASTM penetrations into centipoises, Table II—derived by the author in Part I in accordance with the fundamental equation of Pendleton [6]—was used.

(ii) BRTA viscosities in seconds were multiplied by 394 to give the equivalent kinematic values in centistokes [Pickard [7]].

(iii) Saybolt Furol viscosities were converted to centistokes from tables given in the " Science of Petroleum." [8]

Centistokes multiplied by the corrected specific gravity at the relevant test temperatures gave centipoises. The experimental results and final conversions into absolute units are all given in Table VI, which has been drawn up to cover log–log centipoises over the whole range of temperatures under consideration, viz., from 77° to 302° F.

Discussion

The results shown in Table VI have been depicted graphically in Fig 32, in which log–log centipoises have been plotted against log ° F absolute in accordance with the fundamental straight-line relationship discussed in Part I. For reasons of clarity, only four complete sets of lines have been

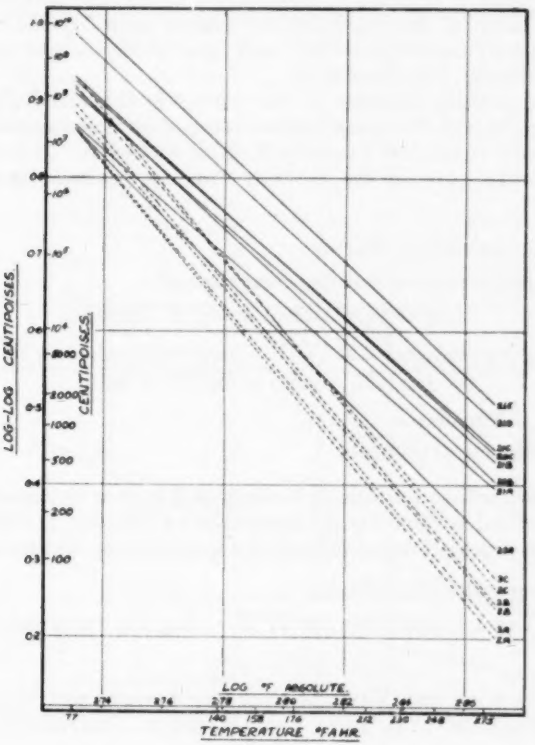

FIG 32

EXPERIMENTAL DATA RELATING LOG–LOG CENTIPOISES WITH LOG ° F ABSOLUTE FOR DIFFERENT CLASSES OF BITUMEN

included in the diagram to demonstrate the essential differences in behaviour

(i) between typical high Viscosity Number classes of bitumens, *e.g.*, Groups 20A–C and 21A–E (shown in full lines) and typical low Viscosity Number groups of bitumens, *e.g.*, Groups 2A–C and 3A–C (shown in dotted lines), and

(ii) between hard and soft members of each group.

The remainder of results in Table VI can also be plotted in the same way, and it will be found that bitumen groups with intermediate Viscosity

Numbers occupy intermediate positions on the chart and have intermediate slopes.

The fact that excellent straight-line relationships have been obtained for each set of results, which included such diverse data as penetrations at 77° F, BRTA viscosities at 140°, 158°, and 176° F respectively, and Saybolt Furol viscosities at temperatures ranging from 212° to 302° F—all converted to absolute units of viscosity by appropriate equations—adds convincingly to the reliability of the important deductions made by the author from Pendleton's work described earlier [1] and upon which the new conception of " True Penetration " is dependent.

The most striking features of the curves is the relatively flat slope exhibited by the high Viscosity Number series of bitumens as compared with the steep slope of the low Viscosity Number materials. It is these slopes which form the basis of the author's Fluidity Characterization Factor. Thus :—

Fluidity Characterization Factor

$$= \text{Negative slope of line depicting data of Log–log centipoises } \textit{vs} \text{ log° F Absolute}$$

$$= \frac{\text{Log–log centipoises at 77° F} - \text{Log–log centipoises at 275° F}}{\log (275 + 460) - \log (77 + 460)}$$

$$= \frac{\text{Numerator}}{\text{Arithmetical Constant}}.$$

This expression can accordingly be simplified further by cancelling out the denominator and multiplying the numerator by 100, say, in order to obtain a convenient practical factor instead of a series of long decimals. Thus :—

Fluidity Characterization Factor

$$= 100 \times [\text{Log–log centipoises at 77° F} - \text{Log–log centipoises at 275° F}].$$

Application of the Various Fluidity Indices and Factors to Experimental Data and Graphical Comparisons

The experimental results shown in Table VI have been used for determining the various fluidity indices or factors by computation or by graphical means with the aid of the relevant charts. It should be noted that for convenience only Viscosity Numbers at 176° F have been used as a reference basis, excepting where no viscosity determinations have been made at that temperature, in which case the Viscosity Numbers reported at 158° F have been used.

A direct comparison of the following methods of classification can now be made for bitumens of different classes or Viscosity Numbers :—

1. H.C.C. Fluidity Index.
2. N–H.C.C. Fluidity Index.
3. Zapata Fluidity Factor.
4. Fluidity Characterization Factor.

Comparison between H.C.C. and N-H.C.C. Fluidity Indices

A comparison between these two indices for the tabulated data under consideration appears at first glance to show only minor variations, perhaps so small as not to have warranted any modification at all (by the author) of the original fluidity index of Holmes, Collins, and Child. This, however, is not entirely the case, though it may seem to be so in the present instance

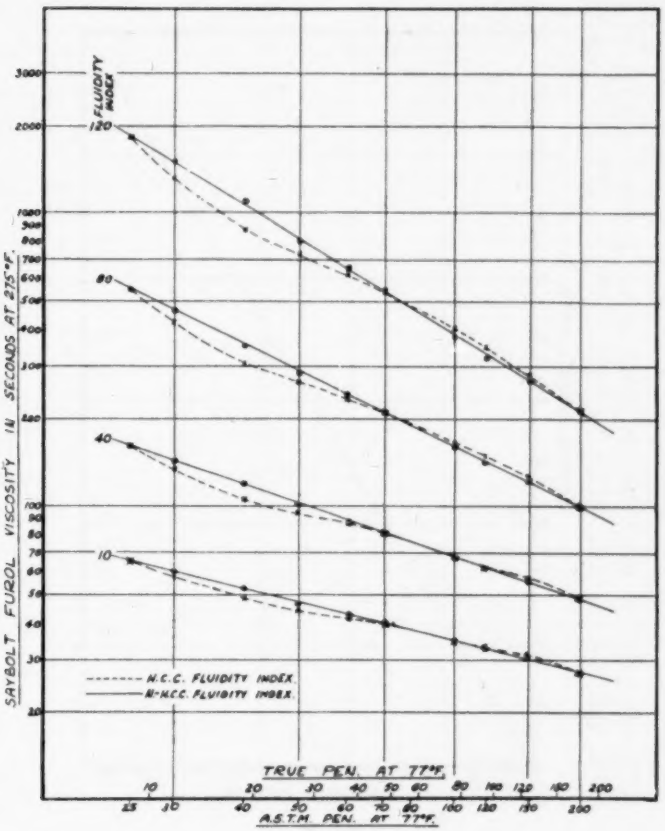

FIG 33

COMPARISON BETWEEN H.C.C. AND N-H.C.C. FLUIDITY INDICES

solely due to the fact that the experimental data of the author (see Table VI) have by chance been obtained in particular for those penetrations at which the deviations have been shown to be relatively small (see earlier discussion on Fig 30).

The point that needs emphasis is that no method of classification can be regarded as being satisfactory unless the principles upon which it is based have a sound foundation. That this is not the case in the present instance

Sample No.	Pen at 77° F	R & B soft. pt, °F	Specific gravities								
			60° F	77° F	140° F	158° F	176° F	212° F	230° F	248° F	275° F
2A	190	98·6	1·079	1·074	1·054	1·049	—	1·032	1·026	—	1·012
2B	100	107·4	1·077	1·072	1·052	1·047	—	1·030	1·024	—	1·010
2C	56	115·6	1·080	1·075	1·055	1·050	1·044	1·033	1·027	—	1·013
3A	180	99·6	1·080	1·075	1·055	1·050	—	1·033	1·027	—	1·013
3B	120	105·0	1·072	1·067	1·047	1·062	—	1·025	1·019	—	1·005
3C	63	114·1	1·082	1·077	1·057	1·052	1·046	—	1·029	—	1·015
5A	185	100·0	1·068	1·063	1·043	1·038	—	1·021	1·015	—	1·001
6A	196	99·2	1·055	1·050	1·030	1·025	—	1·008	—	—	0·988
6B	80	111·9	1·075	1·070	1·050	1·045	1·039	1·028	—	—	1·088
6C	52	118·5	1·069	1·064	1·044	1·039	1·033	1·022	—	—	1·002
7A	200	99·5	1·053	1·048	1·028	1·023	—	1·006	—	—	0·986
7B	83	111·4	1·058	1·053	1·033	1·028	1·022	1·011	—	—	0·991
7C	54	118·0	1·057	1·052	1·032	1·027	1·021	1·010	—	—	0·990
8A	125	106·0	1·036	1·031	1·011	1·006	—	0·989	—	—	0·969
9A	205	100·2	1·050	1·045	1·025	1·020	—	1·003	—	—	0·983
9B	94	110·8	1·060	1·055	1·036	1·031	1·025	—	1·008	—	0·994
9C	54	120·0	1·054	1·049	1·029	1·024	1·018	—	1·001	—	0·987
12A	140	107·0	1·026	1·021	1·001	0·996	0·990	—	0·973	—	0·959
12B	85	115·4	1·045	1·040	1·020	1·015	1·009	—	0·992	—	0·978
15A	210	102·3	1·027	1·022	1·002	0·997	0·991	0·980	—	—	0·960
15B	99	113·0	1·037	1·032	1·012	1·007	1·001	—	0·984	—	0·970
15C	56	121·6	1·047	1·042	1·022	1·017	1·011	—	0·994	—	0·980
15D	27	141·0	1·032	1·027	1·007	—	—	—	—	0·974	0·965
16B	105	112·3	1·026	1·021	1·001	0·996	0·990	—	—	0·968	0·959
16C	57	122·4	1·042	1·037	1·017	1·012	1·006	—	—	0·984	0·975
19A	205	104·3	1·030	1·025	1·005	1·000	0·994	—	—	0·972	0·963
19B	80	121·1	1·030	1·025	1·005	1·000	0·994	—	—	0·972	0·963
19C	55	129·0	1·030	1·025	1·005	1·000	0·994	—	—	0·972	0·963
20A	185	103·5	1·011	1·006	0·986	0·981	0·975	—	0·958	—	0·944
20B	81	120·8	1·027	1·022	1·002	0·997	0·991	—	—	0·969	0·960
20C	55	128·1	1·019	1·014	—	0·989	0·983	—	—	0·961	0·952
21A	190	107·5	1·023	1·018	0·998	0·993	0·987	—	0·970	—	0·956
21B	84	121·1	1·056	1·051	1·031	1·026	1·020	—	—	0·998	0·989
21C	59	126·3	1·030	1·025	—	1·000	0·994	—	—	0·972	0·963
21D	30	143·5	1·036	1·031	—	1·006	1·000	—	—	—	0·969
21E	16	162·2	1·051	1·046	—	—	—	—	—	—	0·984

TABLE VI

Experimental Data for Comparing Various Fluidity Indices

		Viscosity, sec BRTA			Viscosity, sec Saybolt Furol					Eq			
275° F	302° F	140° F	158° F	176° F	212° F	230° F	248° F	275° F	302° F	77° F	140° F	158° F	176
1·012	—	44	13·4	—	163	78·6	—	23·5	—	$2{\cdot}018 \times 10^7$	18,276	5,539	—
1·010	—	100	27·5	—	275	136	—	33	—	$8{\cdot}130 \times 10^7$	41,449	11,349	—
1·013	—	208	52	18	357	184	—	37·5	—	$3{\cdot}388 \times 10^8$	86,457	21,515	7,
1·013	—	49	15	—	176	88	—	25·7	—	$2{\cdot}254 \times 10^7$	20,372	6,206	—
1·005	—	84	24	—	263	126	—	32	—	$5{\cdot}495 \times 10^7$	34,656	9,857	—
1·015	—	185	49	17·5	—	210	—	47	—	$2{\cdot}410 \times 10^8$	77,045	20,314	7,
1·001	—	55	17	—	209	112	—	31·5	—	$2{\cdot}104 \times 10^7$	22,602	6,955	—
0·988	—	56	17·5	—	214	—	—	32	—	$1{\cdot}841 \times 10^7$	22,722	7,073	—
1·088	—	173	48	16	364	—	—	48	—	$1{\cdot}370 \times 10^8$	71,568	19,761	6,
1·002	—	314	76	25·5	520	—	—	53·8	—	$4{\cdot}036 \times 10^8$	129,164	31,108	10,
0·986	—	58	18	—	245	—	—	37·5	—	$1{\cdot}760 \times 10^7$	23,490	7,253	—
0·991	—	175	49	16	365	—	—	49	—	$1{\cdot}225 \times 10^8$	71,225	19,851	6,
0·990	—	319	77	26	506	—	—	55·4	—	$3{\cdot}673 \times 10^8$	129,712	31,159	10,
0·969	—	111	33	—	354	—	—	42·4	—	$4{\cdot}989 \times 10^7$	44,211	13,078	—
0·983	—	72	22·5	—	263	—	—	41·1	—	$1{\cdot}694 \times 10^7$	29,079	9,047	—
0·994	—	192	51	19	—	263	—	60·4	—	$9{\cdot}376 \times 10^7$	78,373	20,713	7,
0·987	—	370	96	32·5	—	352	—	74·7	—	$3{\cdot}673 \times 10^8$	150,008	38,728	13,
0·959	—	188	54	19·5	—	344	—	82·3	—	$3{\cdot}908 \times 10^7$	74,144	21,195	7,
0·978	—	330	87	30·5	—	427	—	92·9	—	$1{\cdot}169 \times 10^8$	132,620	34,794	12,
0·960	—	135	42	18	350	—	—	77	—	$1{\cdot}585 \times 10^7$	53,296	16,500	7,
0·970	—	320	85	31	—	441	—	103	—	$8{\cdot}260 \times 10^7$	127,593	33,724	12,
0·980	—	556	138	47	—	635	—	133	—	$3{\cdot}388 \times 10^8$	223,879	55,294	18,
0·965	—	—	—	—	—	—	684	273	—	$2{\cdot}307 \times 10^9$	—	—	—
0·959	—	295	82	31·5	—	—	245	111	—	$7{\cdot}447 \times 10^7$	116,346	32,181	12,
0·975	—	659	161	55	—	—	340	136	—	$3{\cdot}236 \times 10^8$	264,064	64,191	21,
0·963	—	205	56	20·3	—	—	231	97	—	$1{\cdot}694 \times 10^7$	81,100	22,060	7,
0·963	—	972	255	77	—	—	595	260	—	$1{\cdot}370 \times 10^8$	384,600	100,470	30,
0·963	—	—	364	115	—	—	830	331	—	$3{\cdot}524 \times 10^8$	—	143,420	45,
0·944	—	136	39·3	17·3	—	277	—	69·3	—	$2{\cdot}104 \times 10^7$	52,830	15,186	6,
0·960	—	809	212	68	—	—	438	201	—	$1{\cdot}291 \times 10^8$	326,037	83,279	26,
0·952	—	—	445	123·5	—	—	835	336	—	$3{\cdot}524 \times 10^8$	—	173,401	47,
0·956	—	332	96	40	—	640	—	180	—	$2{\cdot}018 \times 10^7$	130,548	37,555	15,
0·989	—	794	204	77	—	—	679	287	—	$1{\cdot}191 \times 10^8$	322,538	82,470	30,
0·963	—	—	349	116·5	—	—	830	358	—	$2{\cdot}884 \times 10^8$	—	137,510	45,
0·969	0·961	—	1220	364	—	—	—	670	297	$1{\cdot}784 \times 10^9$	—	483,564	143,
0·984	0·976	—	—	—	—	—	—	960	396	$8{\cdot}710 \times 10^9$	—	—	—

TABLE VI

Experimental Data for Comparing Various Fluidity Indices and Factors

Viscosity, sec Saybolt Furol					Equivalent centipoises												
212° F	230° F	248° F	275° F	302° F	77° F	140° F	158° F	176° F	212° F	230° F	248° F	275° F	302° F	77° F	140° F	158° F	176°
163	78·6	—	23·5	—	$2{\cdot}018 \times 10^7$	18,276	5,539	—	362	173	—	45·5	—	0·8636	0·6296	0·5732	—
275	136	—	33	—	$8{\cdot}130 \times 10^7$	41,449	11,349	—	611	300	—	68·7	—	0·8982	0·6645	0·6080	—
357	184	—	37·5	—	$3{\cdot}388 \times 10^8$	86,457	21,515	7,402	797	408	—	80·0	—	0·9309	0·6934	0·6368	0·58
176	88	—	25·7	—	$2{\cdot}254 \times 10^7$	20,372	6,206	—	394	196	—	51·2	—	0·8665	0·6344	0·5789	—
263	126	—	32	—	$5{\cdot}495 \times 10^7$	34,656	9,857	—	581	277	—	66·2	—	0·8887	0·6571	0·6014	—
—	210	—	47	—	$2{\cdot}410 \times 10^8$	77,045	20,314	7,217	—	466	—	101·5	—	0·9233	0·6891	0·6343	0·58
209	112	—	31·5	—	$2{\cdot}104 \times 10^7$	22,602	6,955	—	460	246	—	65·1	—	0·8647	0·6389	0·5845	—
214	—	—	32	—	$1{\cdot}841 \times 10^7$	22,722	7,073	—	464	—	—	65·3	—	0·8612	0·6391	0·5855	—
364	—	—	48	—	$1{\cdot}370 \times 10^8$	71,568	19,761	6,545	809	—	—	103·2	—	0·9105	0·6861	0·6331	0·58
520	—	—	53·8	—	$4{\cdot}036 \times 10^8$	129,164	31,108	10,382	1147	—	—	115	—	0·9349	0·7085	0·6525	0·60
245	—	—	37·5	—	$1{\cdot}760 \times 10^7$	23,490	7,253	—	532	—	—	77·9	—	0·8601	0·6406	0·5867	—
365	—	—	49	—	$1{\cdot}225 \times 10^8$	71,225	19,851	6,445	798	—	—	104·1	—	0·9079	0·6860	0·6333	0·56
506	—	—	55·4	—	$3{\cdot}673 \times 10^8$	129,712	31,159	10,455	1101	—	—	117·5	—	0·9328	0·7087	0·6526	0·60
354	—	—	42·4	—	$4{\cdot}989 \times 10^7$	44,211	13,078	—	757	—	—	87·1	—	0·8864	0·6670	0·6164	—
263	—	—	41·1	—	$1{\cdot}694 \times 10^7$	29,079	9,047	—	569	—	—	85·7	—	0·8590	0·6497	0·5973	—
—	263	—	60·4	—	$9{\cdot}376 \times 10^7$	78,373	20,713	7,677	—	573	—	128·5	—	0·9016	0·6897	0·6351	0·58
—	352	—	74·7	—	$3{\cdot}673 \times 10^8$	150,008	38,728	13,041	—	760	—	158	—	0·9328	0·7140	0·6617	0·61
—	344	—	82·3	—	$3{\cdot}908 \times 10^7$	74,144	21,195	7,603	—	723	—	171	—	0·8803	0·6875	0·6361	0·58
—	427	—	92·9	—	$1{\cdot}169 \times 10^8$	132,620	34,794	12,128	—	914	—	195	—	0·9068	0·7096	0·6547	0·61
350	—	—	77	—	$1{\cdot}585 \times 10^7$	53,296	16,500	7,026	1162	—	—	159	—	0·8573	0·6746	0·6251	0·58
—	441	—	103	—	$8{\cdot}260 \times 10^7$	127,593	33,724	12,222	—	942	—	216	—	0·8986	0·7081	0·6559	0·61
—	635	—	133	—	$3{\cdot}388 \times 10^8$	223,879	55,294	18,724	—	1359	—	270	—	0·9309	0·7284	0·6761	0·63
—	—	684	273	—	$2{\cdot}307 \times 10^9$	—	—	—	—	—	1432	569	—	0·9714	—	—	—
—	—	245	111	—	$7{\cdot}447 \times 10^7$	116,346	32,181	12,286	—	—	513	229	—	0·8961	0·7047	0·6540	0·61
—	—	340	136	—	$3{\cdot}236 \times 10^8$	264,064	64,191	21,800	—	—	723	285	—	0·9299	0·7342	0·6819	0·63
—	—	231	97	—	$1{\cdot}694 \times 10^7$	81,100	22,060	7,925	—	—	485	201	—	0·8590	0·6910	0·6379	0·59
—	—	595	260	—	$1{\cdot}370 \times 10^8$	384,600	100,470	30,200	—	—	1245	540	—	0·9105	0·7470	0·6992	0·65
—	—	830	331	—	$3{\cdot}524 \times 10^8$	—	143,420	45,080	—	—	1725	731	—	0·9319	—	0·7124	0·66
—	277	—	69·3	—	$2{\cdot}104 \times 10^7$	52,830	15,186	6,650	—	573	—	141	—	0·8647	0·6742	0·6213	0·58
—	—	438	201	—	$1{\cdot}291 \times 10^8$	326,037	83,279	26,549	—	—	917	416	—	0·9090	0·7414	0·6921	0·64
—	—	835	336	—	$3{\cdot}524 \times 10^8$	—	173,401	47,810	—	—	1727	691	—	0·9319	—	0·7192	0·67
—	640	—	180	—	$2{\cdot}018 \times 10^7$	130,548	37,555	15,555	—	1336	—	372	—	0·8636	0·7089	0·6604	0·62
—	—	679	287	—	$1{\cdot}191 \times 10^8$	322,538	82,470	30,947	—	—	1460	612	—	0·9073	0·7411	0·6916	0·65
—	—	830	358	—	$2{\cdot}884 \times 10^8$	—	137,510	45,604	—	—	1725	745	—	0·9274	—	0·7108	0·66
—	—	—	670	297	$1{\cdot}784 \times 10^9$	—	483,564	143,180	—	—	—	1399	616	0·9661	—	0·7546	0·71
—	—	—	960	396	$8{\cdot}710 \times 10^9$	—	—	—	—	—	—	2032	835	0·9974	—	—	—

Log–log centipoises						Soft. point number	Viscosity number	H.C.C. fluidity index	N–H.C.C. fluidity index	Zapata fluidity factor	Fluidity char. factor	Poises at soft. pt.
176° F	212° F	230° F	248° F	275° F	302° F							
—	0·4080	0·3500	—	0·2190	—	0·9	0·7	0	1	−316	64·46	12,650
—	0·4449	0·3938	—	0·2642	—	1·1	0·8	9	8	− 67	63·40	12,250
0·5877	0·4626	0·4167	—	0·2795	—	1·0	0·9	4·5	3·5	− 10·4	65·14	13,520
—	0·4142	0·3602	—	0·2327	—	1·2	1·0	3·5	5	−278	63·38	10,620
—	0·4414	0·3878	—	0·2602	—	1·4	1·2	9	8·5	−105·5	62·85	12,050
0·5864	—	0·4263	—	0·3024	—	1·3	1·3	16	15	− 10·2	62·09	12,650
—	0·4254	0·3783	—	0·2585	—	2·2	1·9	15	16	−284	60·62	12,050
—	0·4260	—	—	0·2589	—	2·3	2·4	17	18	−321	60·23	12,050
0·5816	0·4635	—	—	0·3039	—	2·6	2·1	21	19·5	− 25·6	60·66	13,740
0·6037	0·4857	—	—	0·3141	—	2·7	2·4	18	16·5	0·9	62·08	14,420
—	0·4355	—	—	0·2769	—	3·1	2·8	26·5	27	−325	58·32	12,050
0·5609	0·4627	—	—	0·3048	—	2·7	2·3	23·5	21·5	− 28·2	60·31	13,740
0·6040	0·4832	—	—	0·3160	—	3·0	2·9	20	18·5	0·75	61·68	14,420
—	0·4593	—	—	0·2879	—	3·3	3·2	23·5	23·5	−103	59·85	13,060
—	0·4402	—	—	0·2863	—	4·6	4·0	33	34	−335	57·27	10,940
0·5894	—	0·4407	—	0·3240	—	4·0	4·1	34·5	34	− 31·6	57·76	14,190
0·6143	—	0·4597	—	0·3422	—	4·7	4·2	31·5	30	112	59·06	13,300
0·5889	—	0·4561	—	0·3486	—	6·5	6·4	56·5	58·5	− 80·7	53·17	13,520
0·6111	—	0·4714	—	0·3598	—	7·4	6·3	51·5	51	6·7	54·70	12,650
0·5851	0·4864	—	—	0·3426	—	8·2	7·7	67	67·5	−279	51·47	11,480
0·6114	—	0·4733	—	0·3681	—	7·4	7·2	59	60	4	53·05	13,740
0·6306	—	0·4959	—	0·3860	—	6·6	6·6	55	54	43	54·49	15,630
—	—	—	0·4992	0·4401	—	8·7	—	62	60	66·5	53·13	10,620
0·6116	—	—	0·4330	0·3727	—	7·6	7·7	64	65	6·3	52·34	13,740
0·6374	—	—	0·4562	0·3900	—	7·7	7·6	55·5	54·5	45	53·99	15,140
0·5909	—	—	0·4290	0·3622	—	9·5	8·7	80	80	−221	49·68	10,790
0·6513	—	—	0·4907	0·4365	—	12·2	11·5	93	94	144	47·40	13,740
0·6679	—	—	0·5101	0·4570	—	14·6	11·9	94	91·5	163	47·49	15,140
0·5824	—	0·4406	—	0·3322	—	7·2	7·3	56·5	57	−214	53·25	11,120
0·6458	—	—	0·4716	0·4181	—	11·9	10·9	82·5	83	97	49·09	13,960
0·6702	—	—	0·5102	0·4532	—	12·1	12·4	93	90·5	154·4	47·87	15,630
0·6224	—	0·4950	—	0·4100	—	13·0	12·4	108	108·5	− 19	45·36	13,520
0·6523	—	—	0·5003	0·4452	—	12·6	11·7	99·5	99·5	171	46·21	14,660
0·6683	—	—	0·5101	0·4582	—	11·8	12·4	97	95	176	46·92	17,860
0·7123	—	—	—	0·4977	0·4455	12·1	14·7	96	92	192	47·84	15,890
—	—	—	—	0·5195	0·4656	12·4	—	86	—	151	47·79	9,330

[*To face p.* 466.

with the H.C.C. Fluidity Index can clearly be seen from Fig 33, in which log Saybolt Furol Viscosity at 275° F is the ordinate scale and log true penetration the abscissa. For the sake of practical utility the positions of the equivalent ASTM penetrations have also been inserted in the chart. Four sets of data for fluidity indices of 10, 40, 80, and 120 respectively have been included in the diagram to offer a direct comparison between the H.C.C. and the author's modified N–H.C.C. values respectively.

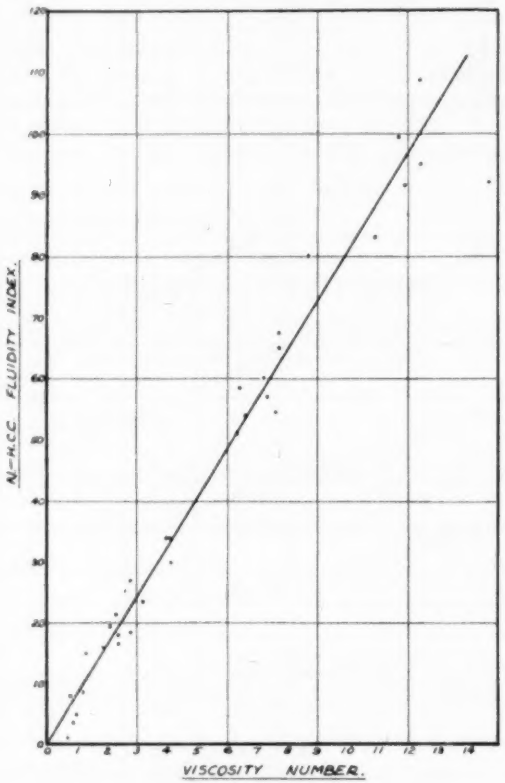

FIG 34

RELATIONSHIP BETWEEN N-H.C.C. FLUIDITY INDEX AND VISCOSITY NUMBER

The requisite viscosity *vs* penetration data for the two types of indices have been obtained directly from Figs 30 and 31 respectively, simply by ruling vertical lines at the desired fluidity indices on each chart and reading off the viscosities corresponding to the various ASTM penetrations at 77° F.

The curves representing the H.C.C. data are most irregular in shape, whereas perfectly straight lines are obtained for the author's modification. This method of depicting the indices is particularly effective in demon-

strating the equality of the indices at the two extreme ASTM penetrations under consideration (namely at 200 and 25 respectively) and also approximately over the intermediate range of 60 to 100 pen with only a fairly slight positive deviation from about 100 to 200 pen. The maximum negative deviation occurs at about 40 pen.

Relationship between N–H.C.C. Fluidity Index and Viscosity Number

The experimental values given in Table VI have been used for plotting Fig 34, which has been constructed to show the relationship between the N–H.C.C. Fluidity Index and Viscosity Number. It appears that within reasonable limits direct proportionality exists. Particularly significant is the fact that the straight-line curve passes through or very close to the origin. This is due to the fact that, during the original construction of the charts, a zero value was in each case arbitrarily assigned to severely cracked bitumens.

Numerically, the N–H.C.C. Fluidity Index is about eight times the corresponding Viscosity Number.

Relationship between Zapata Fluidity Factor and Viscosity Number

The relevant data of Table VI have also been used for the construction of Fig 35, in which Zapata Fluidity Factors have been plotted against Viscosity Number.

In this case it is clear that there is no simple relationship whatsoever between the two methods of classification.

The positions of the curves in Fig 35 show that the highest Zapata Fluidity Factors are obtained with materials of the lowest penetrations in accordance with the author's discussion earlier. It is significant that, for bitumens of a given penetration, there is a definite trend of increasing factors with increasing Viscosity Numbers excepting for materials of low Viscosity Number.

The most important observation, however, is that the Zapata factors for bitumens of about 50 to 60 pen are invariably higher than the corresponding values for materials of about 180 to 200 pen, irrespective of the fact that Viscosity Numbers ranging widely from 0 to 13 are under comparison. It is clear therefore that the Zapata Fluidity Factor is decidedly more dependent on the hardness of a bitumen than on its quality. This method of classification is consequently radically unsound, since the first requisite of a satisfactory index is ability to identify materials of different classes and sources irrespective of their consistency.

In this connexion it must be pointed out that certain American specifications for asphalt cements having penetrations between 40 and 100 call for a minimum limit of 140 for the Zapata Fluidity Factor. That the inclusion of such a stringent requirement in specifications is unjustified is clearly demonstrated by Fig 35. The author's results are not unique in this respect. Lang and Thomas [9] found that only three out of twenty-five different types of samples of 85 to 100 pen passed this test and only two out of eighteen of 50 to 60 pen. Lewis and Welborn [10] too, tested thirty-nine typical bitumens of 50 to 60 pen grade and found that twenty-two of these

failed to meet the minimum requirement of 140. Again, only six out of forty samples of the 85 to 100 grade passed.

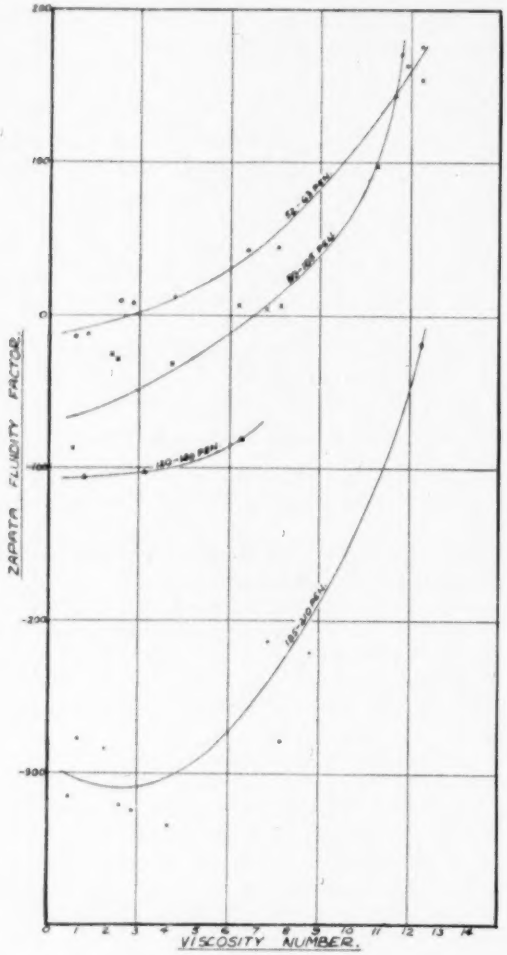

FIG 35

RELATIONSHIP BETWEEN ZAPATA FLUIDITY FACTOR AND VISCOSITY NUMBER

Relationship between Fluidity Characterization Factor and Viscosity Number

Fig 36, based on experimental data given in Table VI, shows that these two methods of classification are related to each other by a straight line graph.

It is noteworthy that the author's Fluidity Characterization Factor differs from the N–H.C.C. Fluidity Index in that, in this case, bitumens of high

Viscosity Number have a low fluidity characterization factor, and the curve has a negative slope. Since, by definition of the author, the fluidity factor is a measure of temperature susceptibility from 77° to 275° F, it follows that the Viscosity Number classification itself also offers a direct measure of the same property.

Fig 36 taken in conjunction with Fig 34 also shows that the N–H.C.C. Fluidity Index and the Fluidity Characterization Factor are directly proportional to each other. N–H.C.C. values of 0 and 100, corresponding to

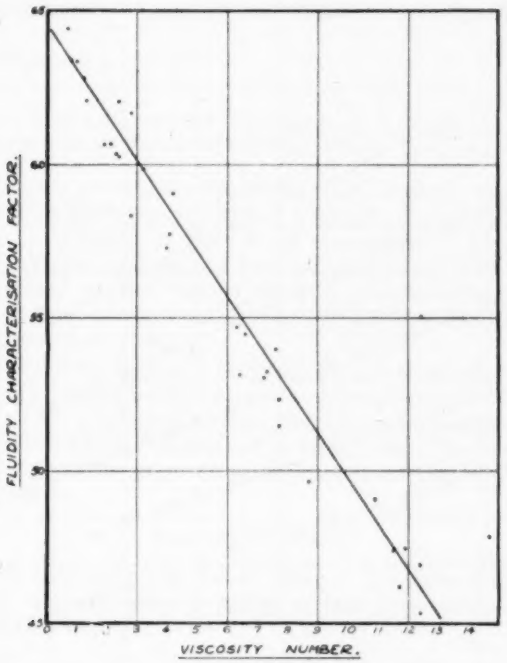

FIG 36

RELATIONSHIP BETWEEN FLUIDITY CHARACTERIZATION FACTOR AND VISCOSITY NUMBER

Viscosity Numbers of 0 and 12·4 respectively, are approximately equivalent to F.C.F. values of 64·5 and 46·2 respectively.

It follows too that a Fluidity Characterization Factor chart can be constructed in a similar manner to Fig 33, so that log viscosity in centipoises at 275° F plotted against log true penetration at 77° F should give a straight line.

Examination of the equation :—

Fluidity Characterization Factor

= 100 [log–log centipoises at 77° F — log–log centipoises at 275° F]

shows that if two of the terms are fixed, then the third can be calculated.

A table (not shown) has consequently been drawn up to determine the centipoises at 275° F corresponding to a series of ASTM penetrations ranging from 200 down to 25 for Fluidity Characterization Factors of 65, 60, 55, 50, and 45 respectively. The penetrations have been converted directly to centipoises by means of Table II in Part I. The viscosities at 275° F have been left in absolute units because further conversion to Say-

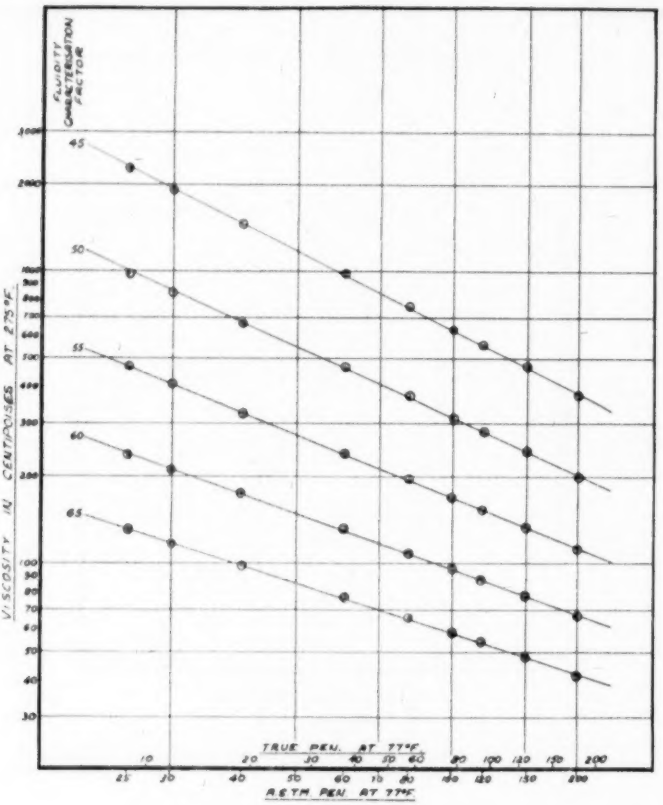

FIG 37

FLUIDITY CHARACTERIZATION FACTOR CHART

bolt Furol seconds necessitates knowledge of the bitumen specific gravity at 275° F, and the latter property is a variable for different classes of material.

Fig 37 has then been constructed with log centipoises at 275° F as the ordinate and log true penetration as abscissa. Index lines are shown for the five standard Fluidity Characterization Factors mentioned above. In each case a perfectly straight line has been obtained, conclusively illustrating that the author's proposed fluidity factor is rational in basis.

This chart can be used directly in practice for determining the Fluidity Characterization Factor of any bitumen, thus avoiding the laborious logarithmic calculations required by the equation. The experimental data required are :—

(*a*) Saybolt Furol viscosity at 275° F. This can be converted to kinematic viscosity in centistokes by means of tables.

(*b*) Specific gravity at 275° F, generally calculated by subtracting a known correction factor from the specific gravity determined at a lower temperature. Value (*a*) multiplied by (*b*) gives centipoises at 275° F.

(*c*) Penetration at 77° F.

Experimental Confirmation of Theoretical Values for Viscosities of Bitumens at their Softening Point Temperatures

Table VI offers a convenient opportunity for checking the correctness of the theoretically derived viscosities of bitumens at their softening-point temperatures, as discussed in Part II of this series of papers. In the present instances, the softening points all occur well within the temperature limits used for the various viscosity determinations, whereas in the previous theoretical derivations (see Fig 15) the results had to be extrapolated from known data.

The various samples included in Table VI have actual viscosities at their respective softening-point temperatures ranging from 9330 poises to 17,860 poises. It is of interest to note that the experimental results confirm the theoretical conclusion reached in Part II that the viscosity at the softening point is not to any extent dependent on the bitumen quality or Viscosity Number.

Acknowledgment

The author is indebted to the South African Torbanite Mining and Refining Co. Ltd., for permission to utilize certain of his experimental data for purposes of this study.

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CORRESPONDENCE

"Acid Sludge—Its Utilization and Disposal."

To the Editor, *Journal of the Institute of Petroleum.*

SIR,

I should like to refer to certain items on the utilization of refinery sludge acids ascribed to Mr Nieuwenhuis on pp. 51 and 54 of the January 1952 issue of the *Journal.*

Mr Nieuwenhuis, in replying to questions regarding performance of the MRW (Monsanto–Ross–Wilde) sludge-acid process in Curacao, made some statements which, although probably entirely correct as far as his information went, apply correctly only to the early days of operation at Curacao. That plant was built by a British firm using British equipment and was completed just at the end of the war. Considerable early difficulties were encountered, and these difficulties, as Mr Nieuwenhuis infers, were largely mechanical. According to reports from Curacao and from people from the U.S.A. who have inspected the plant, all such difficulties have been substantially overcome and the plant is now operating successfully. An expansion is now under way using similar equipment with only minor modifications.

Substantially none of the difficulties encountered at Curacao were met with in the single-unit plant operated by this process in the U.S.A. during 1944 and 1945. Smoother operation here appeared to be due to equipment rather than to operation, although there were some minor matters, such as rate of coke circulation, in which our practice differed from early practice at Curacao.

In regard to the type of sludge usable in MRW equipment, Mr Nieuwenhuis was in error as far as American practice is concerned, although his comments were in line with early Curacao experience. Our single-unit plant during approximately a year and a half of operation handled sludge-acid mixtures ranging from 38 to 91 per cent titratable acidity. The lower strength referred to was a straight pressure distillate sludge acid, and the higher strength was alkylation spent acid. Nearly the whole range between these two strengths was covered, although at the lower end of the range only a few days was devoted to the various strengths merely to make sure that the process would, if necessary, handle any type of sludge acid sufficiently fluid to permit its deposition on the surface of small coke granules.

There are now under construction in the U.S.A. two plants, each of approximately 300 short tons daily capacity, using the MRW process.

One of them is to operate on mixed sludges of about 70 per cent titratable acidity, and the other upon mixed sludges of about 60 per cent titratable acidity. In one case hydrogen sulphide will be burned for make-up acid, and in the other case elemental sulphur, produced from hydrogen sulphide, will be burned for the same purpose. The first of these two plants will be completed by about the end of this year, and as quickly as practicable thereafter detailed operation data will be published.

T. R. HARNEY

Monsanto Chemical Company,
St Louis 4,
Missouri, U.S.A.

March 28, 1952.

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Oilfield Exploration and Exploitation

Geology

1557. Turbidity currents and sediments in North Atlantic. D. B. Ericson, M. Ewing, and B. C. Heezen. *Bull. Amer. Ass. Petrol. Geol.*, 1952, **36** (3), 489.—In 1947 a series of bottom-sediment cores was taken in the North Atlantic by the research vessel *Atlantis* of the Woods Hole Oceanographic Institution. Among these were several cores which contained sediments not at all in accordance with orthodox theories of sedimentation in deep ocean basins. Stimulated by these findings, authors have taken many more cores on subsequent cruises, with the consequence that there is now at the Lamont Geological Observatory a large collection of deep-sea cores. This abundant material from widely scattered areas of the North Atlantic has yielded evidence indicating that transportation of sediment by turbidity currents is a process of the first importance in deep-sea sedimentation.

These cores show that during Pleistocene and Recent time large quantities of sediment of shallow-water origin have been deposited in the deep basins of the North Atlantic. It is inferred that turbidity currents are responsible for the transportation of this material. The effect of such a depression-filling process upon the regional topography is discussed. The effectiveness of turbidity currents is sufficient to explain the origin of the Hudson Submarine Canyon. In support of the discussion the paper is followed by a list of some of the most important cores, with short lithologic descriptions. E. N. T.

1558. Shallow well drilled by Carter in Illinois. Anon. *Petrol. Engr*, Feb. 1952, **24** (2), B96.—2 Carrall, Bull Island, White County, Illinois, on a 2-hr test of the Close sand from 1866 to 1881 ft recovered 120 ft gas, 570 ft clean oil, and 50 ft oil-cut mud. B.h.p. was 661 lb. C. A. F.

1559. Zonation of Upper Cretaceous Austin Chalk and Burditt Marl, Williamson County, Texas. K. Young and E. Marks. *Bull. Amer. Ass. Petrol. Geol.*, 1952, **36** (3), 477.—Biostratigraphic methods applied to the Upper Cretaceous "Austin group" in Williamson County, Texas, show that this group can be subdivided into six faunal zones. These zones have been successfully employed in the detailed mapping of parts of the Georgetown Quadrangle in central and southern Williamson County. The present paper reports preliminary results of a study designed to outline the stratigraphy of the "Austin group" of central Texas.

Correlation with standard Cretaceous Zones is very tentative, but the Burditt marl corresponds closely with the Upper Santonian, and the Austin chalk is Lower Santonian and Coniacian.

Named after characteristic species, the zones are based on faunal suites. Some fossils in each suite are abundant enough for detailed mapping. E. N. T.

1560. Two fields opened in Permian basin. Anon. *Petrol. Engr*, Feb. 1952, **24** (2), B98.—Two new oilfields have been discovered in the Permian basin in Iron County, West Texas.

1 Pearl Wilson, 3 miles south of the Tankersley Wolfcamp field, flowed 311 b.d. from the Strawn at 7230 to 7246 ft.

1-B Noelke flowed 21·7 b.d. from 7413 to 7466 ft. G.O.R. was 5800 : 1. C. A. F.

1561. East Texas interest enlivened by Georgetown find. W. B. Colvin. *World Oil*, Mar. 1952, **134** (4), 74.—The completion of W. T. Bruton 1, Houston County, for 273 b.d. of 43° oil from the Georgetown limestone at 9170 to 9230 ft has stimulated interest in the oil potential of this formation, previously found to be dry.

Although the discovery may be only of local interest, there may be discoveries in the limestone in parts of five counties: Houston, Madison, Trinity, San Augustine, and Sabine.

Future exploration for similar production will require careful control of drilling and completion practice because of the dangers of mudding off and lost circulation in the low-pressure limestone. C. A. F.

1562. Wildcat successful in Escambia, Alabama. Anon. *Petrol. Engr*, Feb. 1952, **24** (2), B96.—A wildcat in Escambia County, Alabama, on test recovered 4650 ft of clean oil in 26 min from 5945 to 5950 ft. 25 ft of pay were found. C. A. F.

1563. Vassen well shows gas at 20,300-ft depth. Anon. *Petrol. Engr*, Feb. 1952, **24** (2), B86.—George Vassen 1, Stone County, Mississippi, flowed rich gas from 20,300 ft. Bottom of hole is 20,450 ft. This is the world's second deepest well. C. A. F.

1564. Kentucky gets new field. Anon. *Petrol. Engr*, Feb. 1952, **24** (2), B96.—1 Mahourin–Luck Community, Webster County, Kentucky, found two pays in the McCloskey limestone which flowed 1000 brl in 21 hr and 512 brl in 24 hr. C. A. F.

1565. Husky–Wilshire opens new area in Wyoming. Anon. *Petrol. Engr*, Feb. 1952, **24** (2), B101.—Torgeson 1, Big Horn County, Wyoming, found 86 ft of gas pay in the Embar, topped at 11,645 ft. Potential was 20,000,000 cu. ft/day containing 14% SO_2. High-gravity oil has also been found. C. A. F.

1566. Olson oil opens new pool in Oklahoma County. Anon. *Petrol. Engr*, Feb. 1952, **24** (2), B98.—2 Erle Deacon "A," Lincoln County, Oklahoma, on test flowed 25 brl/hr from Bartlesville sand from 3280 to 3300 ft. C. A. F.

1567. Western Nebraska oil and gas development. Pt 1. E. C. Reed. *World Oil*, 1.2.52, **134** (2), 67.—The discovery of oil in commercial quantities in Cheyenne County, Nebraska, in 1949 has stimulated exploration in western Nebraska, and several producing areas have recently been discovered.

Details of seven fields discovered within the last two years are given; these are the Southeast Gurley, Huntsman, Dorman, McLernon, Marlow, Johnson, and Dalton fields.

A location map of the oil and gas fields in Western Nebraska and a cross-section and structure map of the Gurley field are included. C. A. F.

1568. Western Nebraska oil and gas development. Pt 2. E. C. Reed. *World Oil*, Mar. 1952, **134** (4), 78.—Much of the panhandle area of western Nebraska forms part of the Julesberg–Denver–Cheyenne basin, which contains relatively thick Cretaceous rocks unconformably overlying Jurassic, Permian, and Pennsylvanian. The Cretaceous is unconformably overlain by continental Tertiary varying from 500 to 1500 ft in thickness.

The Cretaceous consists of, from top to base: Pierre shale, Niobrara, Carlile shale, Greenhorn limestone, Graneros shale, and the Dakota group. Oil is produced from the Dakota, which varies from 450 to 700 ft in thickness. The group consists of sandstones and interbedded shales which were deposited in a transgressive sea which expanded eastwards across Nebraska.

The top of the upper sandstone of the Dakota, which is used as a sub-surface marker, shows the basin to be deepest in western Kimball and southwestern Banner counties. Details of local structure in the basin are known only in the Southeast Gurley field and the Huntsman area in Cheyenne County. The Southeast Gurley field structure trends east–west with an effective closure of approx 60 ft or more. Most oilfields in Cheyenne County occur on a north–northeast trend controlled by favourable sand conditions. Seismic surveys are the best tool for locating favourable structures in the area.

C. A. F.

1569. Nebraska gets 2-zone well. Anon. *Petrol. Engr*, Feb. 1952, **24** (2), B98.—1 Kugler, Cheyenne County, Nebraska, on test of the First Dakota at 4385 to 4390 ft flowed 3,365,000 cu. ft. gas/day and recovered 30 ft condensate. Gas and condensate were also found from 4530 to 4575 ft in the Third Dakota. C. A. F.

1570. Oil and gas in the Uintah Basin of Utah. D. Hager. *Petrol. Engr*, Mar. 1952, **24** (3), A41.—Natural gas has been produced from the Cretaceous Dakota for many years in the Uintah Basin of Colorado and Utah, but the area was not considered to be an important oil and gas reserve untll the discovery of oil in Eocene Green River by Ute Tribal 1 in 1949. Two further fields were discovered in 1950 and 1951, and exploration activity has been intensified.

The basin is a geosynclinal depression covering 15,000 sq. m. lying between the Uintah Mountains to the north, the Uncompaghne uplift to the southeast, the San Rafael Swell to the south, and the Wasatch plateau to the west and southwest. Minor folds project into the basin, generally trending northwest. Formations range from Cambrian to Oligocene with a total sedimentary column of approx 30,000 ft. Surface shows include gilsonite (Uintahite) veins, elaterite or wurtzilite, ozokerite, and asphaltic sands.

The Roosevelt field produces from fractures and joints in Green River shale, and Red Wash and Duchesne, from Green River and Wasatch sands. These pays are less than 15,000 ft in depth everywhere within the basin.

The Green River contains large oil-shale deposits. Source of the free oil in the Eocene beds may be underlying Cretaceous marine shales, or it may be indigeneous.

The three oilfields discovered in the basin are briefly described, and drilling, completion practice, marketing, and refining aspects are outlined.

A geological column, cross-section, and index map of the basin are included.

C. A. F.

1571. Geological history and petroleum possibilities of the Philippines. E. M. Irving. *Bull. Amer. Ass. Petrol. Geol.*, 1952, **36** (3), 437.—Despite numerous indications, the Philippine Archipelago is the only important island group of the great series of Pacific island arcs fringing Asia which has not produced important quantities of petroleum and natural gas. To date, some 164 producing oilfields have been listed as discovered in the Far East since 1873; twelve are on the Asiatic continent, and 152 are within the island arcs, and of the total oil produced more than 85% has come from the island areas.

The geology of the Philippines is not as well known as that of other island groups in this area.

The Philippine Archipelago comprises ten major island areas and a great many small islands in a triangular area between Formosa on the north and Borneo and Celebes on the south. The basement includes mainly volcanic and sedimentary rocks of unknown age, and probably of considerable time range, that have been intruded by ultrabasic to intermediate plutonic rocks as well as by younger volcanics. Fossiliferous Cretaceous, Eocene, and Oligocene sedimentary rocks are found, but it was not until Miocene and Pliocene time that recognizable geosynclinal conditions developed to produce the thick Upper Tertiary sequences now exposed as a result of the late Tertiary and Quaternary disturbances. Certain areas in the Philippines may possess reasonably attractive petroleum possibilities, although exploratory efforts to date have been negative.

E. N. T.

1572. Pemex releases geological information on new fields. Anon. *World Oil*, Mar. 1952, **134** (4), 264.—Geological data are given on five oilfields and one gas field discovered in Mexico in 1951. These are the Jose Colomo field in Tabasco, the Rabon Grande and Concepcion fields in southern Veracruz, the Trevino field and Lomitas gas field in Tamaulipas, and Tamiahua in Veracruz.

Reserves of the Jose Colomo field are over ½ million brl; the structure is an anticline located by seismic reflection, and production is from Miocene Lower Amate sand.

Rabon Grande produces from Miocene Encanto B sand on a salt-dome structure. Reserves are estimated at over 50 million brl.

Trevino produces from Oligocene Frio Trevino sand on an anticline. Gas was also found.

Concepcion produces from Encanto Yucatecal sand on an anticlinal structure due to salt doming.

Tamiahua produces from El Abra limestone; and Lomitas, from Oligocene Vicksburg Lomitas sand on an anticline.

C. A. F.

Geophysics and Geochemical Prospecting

1573. Structural correlation of micromagnetic and reflection surveys. W. P. Jenny. *World Oil*, Mar. 1952, **134** (4), 67.—The development of the airborne magnetometer has made possible the routine application of large-scale micro-magnetics; *i.e.*, the measurement and interpretation of very weak regional and local anomalies which are generally of structural and stratigraphical sedimentary origin.

Practical co-ordination and structural correlation of micro-magnetic and reflection surveys could introduce new aspects into the interpretation of prospects, and four examples of correlation of the two methods are given. It is concluded from these that it is better to correlate the results of two geophysical methods rather than to re-survey the same area by the same method, because the failure of the first survey may not be due to faulty technique or interpretation, but to the inherent weakness of the method. C. A. F.

Drilling

1574. International standardization of oilfield equipment. C. A. Young. *Petrol. Engr*, Jan. 1952, **24** (1), B41.—The work of standardization on pipe threads and other petroleum equipment is summarized and the value of standardization emphasized. C. G. W.

1575. Report on air drilling experiments. R. Sneddon. *Petrol. Engr*, Jan. 1952, **24** (1), B49.—Experience to date is summarized in drilling the Spraberry formation, using air as a circulating medium.

Penetration rates and removal of cuttings have proved satisfactory when using reverse circulation. The major difficulty has been plugging of drill collars and bit. Attempts to avoid this by using a skirted bit have been made, but results are not available. Quantities of air required, penetration rates obtained, and safety measures adopted are discussed. C. G. W.

1576. Spraberry core data discussions. V. B. Ogden and J. Locke. *Petrol. Engr*, Feb. 1952, **24** (2), B55.—General characteristics of the Spraberry formations are permeability 0·002 to 2·5 mD, porosity 6 to 18%, connate water 25 to 45%, residual oil 15 to 25%.

A typical coregraph of the Spraberry Trend is presented, giving permeability, porosity, and fluid-saturation values.

Production is dependent upon fractures existing in the sand body. Calculations indicate that if no fractures existed max possible production would be 5 to 10 b.d. Fractures extending through the well bore to a distance of about 100 ft can increase this max by factors of 100 to 200. C. G. W.

1577. Spraberry sand completion methods. H. E. Legrande. *Petrol. Engr*, Feb. 1952, **24** (2), B48.—1. *Operations before Cementing.*—To prevent contamination of the producing formations by mud, lost circulation materials, and cement two methods have been adopted: (*a*) the hole is plugged with a plastic; (*b*) casing is run without float collars, etc., to prevent excessive pressure surges during running.

2. *Operations after Cementing.*—The trend towards cable tools for drilling in is giving place to reverse circulation using Spraberry crude oil. Compressed-air circulation has been tried, and there is now a tendency to try carefully prepared water-base muds.

Shooting, Hydrafrac, and acid treatments are all in general use.

Well-head equipment is generally standard. C. G. W.

1578. Hydrafrac—the Spraberry stimulant. W. L. Grossman. *Petrol. Engr*, Feb. 1952, **24** (2), B36.—Application of Hydrafrac has been an important factor in advancing the exploitation of the Spraberry sand.

Methods of application in use and their effect on completion operations in this zone are briefly mentioned. C. G. W.

1579. Stratafrac completions. J. M. Moore and R. S. Ousterhoot. *Petrol. Engr*, Feb. 1952, **24** (2), B73.—Stratafrac process consists of pumping a kerosine–acid gel containing 40-mesh sand into the formation. The gel reverts of its own accord to a solution and flows from the formation into the hole, leaving the propping agent behind.

The acid constituents of the gel tend to dissolve calcareous-matter mud and petroleum residues and cement.

Field results of Stratafrac treatments are given. C. G. W.

1580. A test procedure for completion fluids. V. B. Zacher. *Petrol. Engr*, Jan. 1952, **24** (1), B7.—Emulsion muds are briefly discussed. A method of evaluating the suitability of drilling fluids for well completions is then presented.

This consists in making tests of wall thickness, fluid loss, and oil production on cores from the producing interval.

The equipment used is a small core-holder made to fit a 100-lb mud filter-press. The instrument is illustrated and described. Procedure is as follows: (1) extract core; (2) saturate with connate water; (3) flush with kerosine; (4) subject core to 100 lb mud pressure in filter-press; (5) reverse core, flush with crude oil to determine rate of oil production.

Results obtained with a number of drilling fluids indicate that with cores of permeability 400 to 600 mD productions are smaller than some cores of 200 to 300 mD permeability. This is due to the fact that the pore size of the more permeable cores is sufficient to take the finer mud particles, with subsequent plugging. This may be remedied by using muds containing an inert matrix-forming material or by using oil-soluble additives.

The experimental method outlined enables the best possible completion fluid to be selected, necessary changes in fluid properties to be indicated, and the best washing and surveying fluids to be used.

The necessity for further work is emphasized. C. G. W.

1581. Swinging final drive. T. M. Cone. *Petrol. Engr*, Jan. 1952, **24** (1), B31.—This new form of engine drive enables draw-works and engines to be transported without disconnecting the driving-chain.

The draw-works input drive is pivoted at No. 1 engine. A flexible coupling at the draw-works input shaft is disconnected and the drive swung to a vertical position and locked. Draw-works and engine units are then separate, and may be moved to a new location.

Applications are described. C. G. W.

1582. Development of an API thread compound. M. G. Mayberry. *Petrol. Engr*, Jan. 1952, **24** (1), B56.—Requirements of a thread compound are: (1) adequate lubricating properties; (2) stable up to 300° F; (3) must not become fluid at temp up to 300° F; (4) leak resistant at 300° F; (5) must not absorb water; (6) must contain an inert filler to prevent leakage; (7) readily applicable in cold weather.

Tests are described on tubular goods—including pressure testing to 10,000 p.s.i. and the results summarized.

It is concluded that mixtures of lead, zinc, copper, and graphite dispersed in a suitable medium form a permanent leak-resistant seal with adequate lubricating properties. Although not yet complete, tests indicate that the promising compositions contain the following: flake copper 2 to 4%, graphite 18%, powdered lead and zinc 42 to 44% (2·35 to 3·17 lead : 1 of zinc) dispersed in 34 to 36% of silicone-base medium. These proportions may be modified within wide limits, the properties of the compound varying with the composition.

Tests with petroleum-base mediums are not complete. C. G. W.

1583. Drilling costs. D. Siskind. *Petrol. Engr*, Jan. 1952, **24** (1), B14.—Detailed expenditures for drilling contractors and company owners are presented.

The survey covers costs of drilling new wells from the time access roads are constructed until the well is completed and drilling equipment removed.

The results are expected to be of value in making cost estimates and in reducing costs. C. G. W.

1584. Patents. U.S.P. 2,578,888 (18.8.47; 18.12.51). H. H. Kaveler, assr to Phillips Petroleum Co. Lightweight drilling mud comprising sufficient inert, finely divided solids to form a filter-cake on the wall of the well in an oil-in-water emulsion stabilized with an alkali salt of an acidic cellulose derivative.

U.S.P. 2,579,453 (31.1.47; 25.12.51). E. E. Post and W. N. Oldham, assrs to American Cyanamid Co. Drilling fluid comprising an aq clay dispersion and a cold-water-soluble condensation product of quebracho and formaldehyde to control water loss. V. P. P.

Production

1585. Fluid saturation in porous media by X-ray technique. A. D. K. Laird and J. A. Putnam. *Petrol. Tech.*, Oct. 1951, **3** (10), *AIMME Tech. Paper No.* 3169, 275–84.—The theory of the use of X-ray-absorption techniques for measuring fluid saturations in porous media is given. The apparatus and experimental method are described in detail, together with the method of calibration. Several different fluids were used in the calibration, and curves relate meter readings and brine saturation for several cores.

A method for determining the saturations in a three-fluid system has been shown to be theoretically feasible, but requires additional development to place it on a practical basis. G. D. H.

1586. Note on the inter-relationship between wetting and non-wetting phase relative permeability. M. R. J. Wyllie. *Petrol. Tech.*, Oct. 1951, **3** (10), 17.—The wetting-phase relative permeability is given as :

$$K_{rw} = \frac{1}{I^2 S_w^2} \frac{\int_0^{S_w} dS_w/P_c^2}{\int_0^1 dS_w/P_c^2}$$

and for the non-wetting phase the relationship would be :

$$K_{rnw} = \frac{1}{I_n^2 S_0^2} \frac{\int_{S_w}^1 dS_w/P_c^2}{\int_0^1 dS_w/P_c^2}$$

Consequently

$$K_{rnw} = \frac{1}{I_n^2 S_0^2} (1 - K_{rw} I^2 S_w^2)$$

S_w and S_0 are, respectively, the wetting and non-wetting phase saturations, P_c the capillary pressure, I the wetting-phase electrical-resistivity index, and I_n the analogous non-wetting phase-resistivity index. Logarithmic plots of the sparse suitable experimental data give $I_N = S_0^{-1.72}$ in one case and $I_n = S^{-1.87}$ in a second case. G. D. H.

1587. Interstitial water determination by an evaporation method. E. S. Messer. *Petrol. Tech.*, Oct. 1951, **3** (10), *AIMME Tech. Paper No.* 3076, 269–74.—The procedure consists of allowing the saturant in the pore spaces to evaporate until only an irreducible volume remains. The process of evaporation is followed by weighing, and the critical volume is determined either graphically or by a mathematical comparison of fluid flows. The principle of the method is discussed. The time required with water is about 1 hr ; for more volatile liquids, such as benzene or toluene, the time was about 20 min, and a volume factor permits the calculation of the equivalent irreducible water saturation. The characteristics of the sample are unaltered by the test. The method can be used with irregular samples and small core chips. Since the sample tends to become oil-wet after cleaning, the solid surfaces need not be changed, since such organic liquids as toluene will wet the solid with approx zero contact angle.

Results obtained by the use of the method are given, and the means of calculating the capillary pressure from the evaporation curve is presented. A comparison is made between calculated and experimental values. G. D. H.

1588. Report on the Mamou field pressure maintenance project. W. O. Crego and J. M. Henagan. *Petrol. Tech.*, Oct. 1951, **3** (10), *AIMME Tech. Paper No.* 3192, 263–8.—The two main Wilcox oil horizons at Mamou, Louisiana, are the Morein and Deshotels at 11,500 and 11,700 ft subsea, respectively. The former is a blanket sand, broken by a northwest–southeast fault. The east segment has a strong water drive ; the west segment has water injection for pressure maintenance. In 1947 there were nineteen producers and six dry holes. Fresh water reduced the sand permeability,

so a salt-water-injection system was designed. Injection began in the north in early 1949, water being obtained from a sand at 2300 ft in a dry well and mixed with produced water before filtering. The system is closed. For an input of 2000 b.d. there is a 3600 p.s.i. pressure differential at the sand face.

The average sand porosity is 19·7%, average permeability 51 mD, and interstitial water content 35%. There is 80-acre spacing. The solution G.O.R. is 4000, and in 1946 the pressure dropped 1 p.s.i. for 350 brl of produced oil. The initial bottom-hole pressure was 6788 p.s.i. at 11,428 ft subsea. To the end of 1948 801,312 brl of oil had been obtained for a pressure drop of 1705 p.s.i. The bubble point for a subsurface sample was 4527 p.s.i.g.

In 1949 the edge-water rise agreed closely with expectations.

Three months after beginning water injection the pressure decline was nearly arrested. The wells along the south flank of the fluid are giving progressively higher percentages of water. It is expected that water injection will give about 66% more oil than would have been obtained without it, and without decreasing the rate of extraction.

G. D. H.

1589. Reservoir performance and oil properties in Spraberry fields. J. R. Brack. *Petrol. Engr*, Feb. 1952, **24** (2), B77.—Statistical data on Spraberry fields are tabulated.

Typical reservoir fluid properties are also presented.

Reservoir temps of 138° F formation volume factor, 1·4 brl reservoir oil/brl stock-tank oil. Dissolved G.O.R. 725 ft³/brl and API gravity 35·6° are mean values generally applicable. C. G. W.

1590. History of gas lift and its modern application to the petroleum industry. H. V. Eicher. *Petrol. Engr*, Jan. 1952, **24** (1), B36.—Factors important in gas-lift operation and improvements which have enabled gas lift to be applied to wells of low productivity are discussed. C. G. W.

1591. Diluent injection aids heavy oil lift. W. T. Rintoul. *Petrol. Engr*, Feb. 1952, **24** (2), B42.—In one California field only four of the field's 350 wells produce oil with a gravity as high as 16° API.

Thirteen wells in this field produce oil of gravity 6° to 9° API, and difficulty has been experienced in lifting this oil.

By injecting an oil of 21° API gravity, pumped from another field, 850 b.d. of heavy oil can be pumped—about 13% of the field's production. About 25 to 33% of the well output is the crude-oil diluent. The pumping units used are 640,000 inches/lb air-balanced type, operating with a stroke of 10 ft at 3½ to 4 s.p.m.

Wells are pulled and serviced every sixty to ninety days. C. G. W.

Oilfield Development

1592. Exploration off to fast start. Anon. *World Oil*, Mar. 1952, **134** (4), 84.—874 exploratory wells were completed in the U.S.A. in Jan. 1952, compared with 1036 in the previous month. 149 wells were producers.

Tables show the results of exploratory drilling in the U.S.A. in Jan. 1952, by states, and a summary of the results of exploratory drilling. C. A. F.

1593. Preliminary geological and engineering data Wilshire–Ellenburger field, southwest Texas. M. A. Colligan and O. H. Berry. *Petrol. Engr*, Feb. 1952, **24** (2), B7.—The Wilshire–Ellenburger field in southwestern Texas was discovered in 1951 by McElroy 1, and subsequent development was rapid.

The field lies east of the axis of the Midland Basin; the structure may be faulted parallel to the steep southwestern flank by a northwest–southeast normal fault. Pay is Ellenburger dolomite, which is close and even textured; there may be several irregular zones of porosity. Oil–water level is at 9719 ft subsea, and the producing oil column is 541 ft. Initial b.h.p. was 5386 p.s.i. at 9500 ft subsea. Original fluid was undersaturated with satn pressure 1805 p.s.i. Productivity indices have ranged from 1·58 to 3·97 at 377 to 2185 b.d.

Sixteen rigs are operating in the field; major drilling problems are crooked hole, chert, and water supply. Crooked-hole problems, prevalent on the western flank of the structure, may be due to faulting or to stratigraphical differences. Water, originally brought in by truck, is now partly supplied by shallow wells. C. A. F.

1594. 21 wells now completed in new Saskatchewan field. Anon. *World Oil*, Mar. 1952, **134** (4), 264.—Twenty-one wells have been completed for production in the Coleville field, near Kindersley, Saskatchewan. Two of these produce gas.

Royalite Canada Southern 2 was completed in the Viking, and initially produced 7 million cu. ft. gas/day. C. A. F.

1595. Oil in Turkey. K. Kruger. *Petroleum*, Apr. 1952, **15** (4), 89–91.—Describes the geological features of Turkey, with particular reference to oil potentialities, and gives details of the areas which are regarded as being most hopeful for future exploration. R. E. P.

1596. Pyrenean petroleum. A. Coulaty. *Bull. Ass. franç. Tech. Pétrole*, 1952 (91), 3–22.—Summary of activities in Southwest France, illustrated by plans and diagrams. Companies concerned are Régie Autonome des Pétroles (R.A.P.), operating the St Marcet gas field, Société Nationale des Pétroles d'Aquitaine (S.N.P.A.), operating the Lacq oil field, Société Nationale des Pétroles du Languedoc Méditerranéen (S.N.P.L.M.), Société des Pétroles de la Garonne (S.P.G.), and Standard Française des Pétroles (S.F.P.). S.N.P.L.M. has a small producing well in the Camargue district; operations of remaining two companies are still in the preliminary stage. V. B.

Transport and Storage

1597. Breaks in pipeline river crossings—and how to prevent them. P. H. Timothy. *Oil Gas J.*, 10.3.52, **50** (44), 108.—Failures in twenty-six pipeline crossings of the Mississippi at Filter Bend are discussed. The conclusions reached are that constantly varying conditions indicate necessity for flexibility in design, a large-dia line is inflexible, and must be buried beyond possible erosions. The degree of uncertainty of scour data is directly proportional to probability of failure; and dredging at the Cottonwood site on the Mississippi could not be deep enough to bury the pipe beyond scour and erosion action. G. A. C.

1598. Transco's deep marine crossing at Arthur Kill. R. Crowe. *Oil Gas J.*, 31.3.52, **50** (46), 101.—Substantial economies for installation of pipeline crossing of a heavily trafficked waterway were achieved by Transcontinental Gas Pipe Line Co. Rotation of floating bent sections of 24-inch pipe from horizontal to a vertical position was accomplished by removal of pontoons. Step-by-step procedure is given. G. A. C.

1599. Pumps. G. L. Farrar. *Oil Gas J.*, 7.4.52, **50** (48), 131.—Pt 1 of this section deals with the pump and the pumping system; equations are given and the solution of an actual pumping problem worked out.

Pt 2 concerns the pumping of volatile liquids, and a graph gives data on suction lifts and suction pressure. G. A. C.

1600. Increasing packing life. E. L. Spence. *Chem. Engng*, Feb. 1952, **59** (2), 200–1.—Practical points in storing, installing, and lubricating packings are given to increase their effective life. D. H.

1601. Patent. U.S.P. 2,576,167 (2.5.47; 27.11.51). L. P. Zick, assr to Chicago Bridge & Iron Co. Fluid storage tank stressed against internal pressure by means of a ring compression member A attached to the cylindrical side walls and a tension member in the shape of a catenary attached to A, to the bottom of the tank, and to vertical tie members fixed to stiffeners in the bottom of the tank. V. P. P.

Refinery Operations

Refineries and Auxiliary Refinery Plant

1602. Tennessee Gas begins operation of refrigerated extraction plant. J. J. King and R. V. Mertz. *Refiner*, Mar. 1952, **31** (3), 118–24.—A description is given of a 75,000 million-cu. ft/day plant processing natural gas recently completed by the Tennessee Gas Transmission Co. Natural gas is dehydrated in eight 20-ft-deep beds of alumina granules, and is then cooled to −104° F at 535 p.s.i.g. in four heat-exchanger trains, where about 5·5 mol-% of the gas condenses. The condensate is flashed twice at successively lower pressures and fractionated to remove methane and control the proportion of ethane in the product. A flow diagram and material balances are given. A. R. H.

1603. Flame stack design whips wind problem. Anon. *Petrol. Process.*, Mar. 1952, **7** (3), 355–6.—To ensure ignition of the main gas stream, the flare-tip in use at the Salt Lake Refining Co's refinery has six pairs of pilot lines and pilot burners, spaced evenly around the periphery of the stack-top. A drawing of the flare-stack tip is included. D. W. F.

1604. New Dunkirk refinery. V. S. Swaminathan. *Petrol. Engr*, Mar. 1952, **24** (3), C39–43.—The rebuilding and expansion of the Dunkirk refinery has been carried out by concentrating units from both Dunkirk and Cowchelettes refineries to give a plant capacity of 29,000 b.d. crude. Details are given of the rebuilding and of the operations of the rebuilt refinery. E. K. J.

1605. Design of gasoline plants. Pt 4. The arrangement of the elements of the piping system. O. L. Lewis. *Petrol. Engr*, Mar. 1952, **24** (3), C5–10.—(See Abs. 956.) Pt 3 of this series dealt with the general principles involved in the piping layout which are extended in this article to the positioning of the details of the pipe-work. The location and construction are described in detail for: (1) control valves; (2) orifice flanges (3) thermo-wells; (4) pressure gauges; (5) steam traps; (6) gauge glasses; (7) vents and drains; (8) level controls; and (9) valves. Diagrams show the various arrangements of the first six of the above items, emphasizing the most desirable set-ups.

Tables accompanying the diagrams give recommended dimensions for location and construction of orifices, thermo-wells, and gauge glasses. The condensation from saturated steam lines due to radiation to a 70° F room temp can be obtained from a chart. E. K. J.

1606. Centrifugal separation of solid matter and fluids. P. van Riel. *Ingenieur*, 29.2.52, **64** (9), Ch11.—Short introductory paragraphs cover the history, limitations, and function of such equipment, and point out the difference between centrifuges and separators. Then follow, with diagrams and photographs, descriptions of every type of machine in general industrial use. A table giving the capacities of various centrifuges is included. R. R.

1607. Industrial separators, mainly for the continuous separation and clarification of liquids. R. C. Nauta. *Ingenieur*, 7.3.52, **64** (10), Ch23.—Deals in some detail with history of centrifugal separators, and concentrates on the two types of separation, viz., clarification and purification, which are defined respectively as follows: clarification is the separation of solids from a single liquid, while purification is the separation of two liquids of differing specific gravities, one of these liquids carrying with it a small amount of solid material. The application of the various types of centrifugal separator is thoroughly explained, and numerous illustrations of these machines are included. In conclusion, there is a short reference to hydrocyclones. R. R.

1608. Chlorination of condenser cooling water. C. J. Pulham. *Ingenieur*, 28.3.52, **64** (13), W11.—Points out how bacterial organic matter can act as a binder for materials of non-organic character, such as carbonate scales. The bactericidal action of chlorine is stated to be due to its hydrolysis to HOCl, which has the ability to diffuse

through the cell membranes much more readily than other conventional oxidizing agents. It is pointed out how the presence of ammonia or organic nitrogen compounds can reduce the oxidation potential of the chlorine, and therefore its ability to sterilize. The methods of overcoming this are dealt with in great detail. R. R.

1609. Application of the measurement of dielectric constants of suspensions in the study of decantation. R. Jottrand. *Chem. Engng Sci.*, 1951, **1** (2), 81–5.—A method by means of which the concentration of a suspension at any point is determined by measuring the capacity of a condenser designed so as not to interfere with the effect being studied. Experiments using silica powder (56 μ dia) in a benzene/nitrobenzene mixture indicate that the degree of packing of the sediment is a function of its height and not of the time it is left in the settling tank. The method can also be employed for measurements in cyclone separators or in fluidized beds. S. B.

1610. Some notes on seals for rotating and reciprocating shafts. E. F. Boon. *Ingenieur*, 21.3.52, **64** (12), Ch33.—Various formulæ are given for calculating the fluid flow and heat generation through seals. The min out-of-balance for a mechanical seal is calculated, amounting to 50% for liquid flow and 66% for gas flow through the seal for zero spring pressure.

It is shown that contact between the seal faces cannot be avoided. The theory of Brkich, which assumes that the surface tension can prevent this direct contact, is not accepted, because the shape of the interface at the downstream side of the seal as assumed by Brkich is highly improbable. As an example, formula are used to calculate the leakage of a seal fitted to a high-pressure compressor and of a Flexibox mechanical seal. R. R.

1611. Valves and drums as good as new. C. N. Vallette and D. P. Thornton. *Petrol. Process.*, Mar. 1952, **7** (3), 340–7.—The salvaging of valves, flanges, and drums is performed on a production-line basis at the Houston refinery of Sinclair Refining Co. The procedure for valve reconditioning, which is described, involves primarily the sorting into size groups, each group accumulating between thirty and sixty valves before being taken to the shop for repairing. Valves requiring least attention are handled first and sent to storage. Each batch is completed before another is commenced. Flanges are similarly treated. Sketches and descriptions are given of two special jigs used for refacing gate-valve seat rings and wedge refacing. Another department attends to drum reconditioning, in which hydraulic de-denting and chime sealing and straightening are part of the procedure. Drums can be reconditioned as new at the rate of 650 to 700 per 8-hr day. Since reconditioned items are used instead of new, while available, the salvage department is able to show a considerable saving, though not an actual profit, as reconditioned goods are not charged to units.

D. W. F.

1612. Thermal insulation for industrial requirements. R. Thomas. *Refiner*, Feb. 1952, **31** (2), 117–21; Mar. 1952, **31** (3), 143–8.—(See Abs. 1389.) The design of insulation for high-temp range is discussed in Pt II, with special reference to the characteristics of insulation and accessories. Multi-layer insulation is desirable wherever 3 inches or more of insulation is justified, since losses at joints can be reduced by staggering.

In Pt III the specification and application of high-temperature heat insulation for chemical plant are discussed. A. R. H.

1613. Heat transfer and pressure drop in fixed beds of spherical and cylindrical solids. Pt II. J. M. Campbell and R. L. Huntington. *Refiner*, Feb. 1952, **31** (2), 123–31.—(See Abs. 1022.) Data are presented on rates of heat transfer to and from a variety of commercial packings contained in 2, 4 and 6 inch dia columns. Overall coefficients are correlated by the equation :—

$$\frac{U}{C_p G} = 0{\cdot}76\,\epsilon^{-0{\cdot}0225}\,aD_i\left(\frac{G}{a\mu}\right)^{-0{\cdot}42}$$

where U = overall coefficient based on the inside tube wall area and the temp difference between the centre of the bed and the wall; G = mass velocity; C_p = specific heat

of gas; a = effective area of heat transfer per unit volume, D_t = tube dia; μ = visc of the gas. The equation is applicable when the value of the Prandtl group is near 0·80. Temp gradients were determined in radial and axial directions and correlated by the equation :—

$$\frac{K}{kg} = 10{\cdot}0 + 0{\cdot}267\left(\frac{G}{a\mu}\right)$$

K = overall fluid–solid effective thermal conductivity; kg = thermal conductivity of fluids. It is concluded that heat transfer takes place mainly by convection, and free convection at zero mass velocity accounts for about 50% of the transfer at zero mass velocity. A. R. H.

1614. Vacuum truck proves versatile tool. S. J. Artese. *Oil Gas J.*, 31.3.52, **50** (47), 82.—The truck consists of a tank on an ordinary truck chassis, and a motor-driven compressor between tank and truck.

This is used by Shell Oil Co. at Wilmington, Calif., for cleaning drain-boxes, cooling towers, and removing tank sediments. Care must be taken to avoid foaming with certain materials, and light products should not be handled. G. A. C.

1615. Plant radio "network" saves time. D. P. Thornton. *Petrol. Process.*, Feb. 1952, **7** (2), 178–80.—Continuous radio contact is kept with several maintenance trucks on the Torrance, Calif., refinery of General Petroleum Corp. The central transmitter at the maintenance department enables messages to be passed on to maintenance crews whilst still out in the refinery. It is a frequency modulated set, and is identical to the mobile units except for power-supply and auxiliary equipment. Power requirements are mentioned. The range of each set is about 20 miles, giving good reception anywhere within the refinery. D. W. F.

1616. Liquid industrial wastes. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 467–552.—A symposium by various authors, including the following: Antibiotics, J. M. Brown and J. G. Nierdercorn, 468–72; Grain distilleries, G. S. Boriff, 491–3; Organic chemicals manufacture, R. W. Hess and C. J. Carney, 494–8; Petroleum refineries, N. J. Gothard and J. A. Fowler, 503–7; Removal of colour in wastes, W. Rudolfs and W. D. Hanlon, 531–3; Sulphuric acid recovery from waste liquors, F. J. Bartholomew, 541–4. E. J. C.

1617. Patent. U.S.P. 2,576,228 (30.7.49; 27.11.51). W. R. Kinnaird, assr to Universal Oil Products Co. High-temp reactor comprising an elongated pressure-tight chamber A provided at one end with a pair of opposite inlets opening into a mixing member having a restricted throat transverse to the inlets and a flared outlet section discharging axially into A, ignition means extending into A, and product outlets from A. V. P. P.

Distillation

1618. Hydrocarbon extraction by refrigeration. J. J. King and R. V. Mertz. *Oil Gas J.*, 10.3.52, **50** (44), 95.—The Greensburg, Ky, plant of the Tennessee Gas Transmission Co. is described.

Natural gas from the Company's pipeline is treated by refrigeration to remove ethane and heavier hydrocarbons, the stripped gas being returned to the line. Capacity is 750,000 M.c.f. per day. The entire gas stream is chilled to −96° F in the process; certain temps reach −121° F.

The extraction plant removes 380,000 gal daily of heavier hydrocarbons. Thirteen 1760-h.p. compressor units are used for process and refrigerant operations. Flow sheets illustrate the article. G. A. C.

1619. Wetted-wall tube-plate column. J. L. Martin. *Industr. Engng Chem.*, Apr. 1952, **44** (4), 920–4.—Apparatus and experimental results are reported for improving the efficiencies of mass transfer between gas and liquid phases. Preliminary results suggest that the wetted-wall tube-plate column is better than conventional types, but further research is necessary to investigate such variables as tube dia and liquid visc. E. J. C.

1620. Vapour–liquid equilibrium relationships—propane–propylene system. G. H. Hanson, R. J. Hogan, W. T. Nelson, and M. R. Cines. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 604–9.—Vapour–liquid equilibrium data are reported for the system propane–propylene over the range 25° to 135° F at 332·5 p.s.i., which is suitable for use in the design of fractionating towers for high-purity recovery of propylene. The data is graphically correlated with data published by Reamer and Sage (*ibid.*, 1951, **43**, 1628).

Activity coeff are tabulated for both components up to 600 p.s.i. as well as fugacity–pressure ratios for both up to 500 p.s.i. E. J. C.

1621. Vapour–liquid equilibria at sub-atmospheric pressures—system dodecane–hexadecane. J. R. Keistler and M. van Winkle. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 622–4.—Vapour–liquid equilibrium data are reported for the above system at 10, 20, 50, 100, 200, 400, and 760 mm Hg. E. J. C.

1622. Methods for treating data from multiplate equilibrium stills. T. M. Reed and H. S. Myers. *Industr. Engng Chem.*, Apr. 1952, **44** (4), 914–16.—Construction of X–Y diagrams from vapour–liquid equilibrium data obtained by multi-plate columns is not always obvious, as the samples may differ by more than one equilibrium plate. Two graphical methods are described to overcome this. The first depends upon the taking of three samples at points in the column separated by more than one equilibrium plate. The second method relies on two sample points and at least one known point on the conventional equilibrium curve. Two methods permit accurate values of relative volatility to be assigned to any intermediate composition between the sample-point compositions. E. J. C.

1623. Laboratory continuous stills—apparatus and operation. A. C. Wilcox, K. E. Coulter, and L. E. Lloyd. *Refiner*, Feb. 1952, **31** (2), 134–6.—Continuous laboratory stills and their operation are discussed. A. R. H.

1624. Vacuum distillation plants for petroleum. E. Reisemann. *Erdöl u. Kohle*, 1952, **5**, 161–7.—Review, mainly of U.S. developments and largely based on paper by Kraft (see Abs. 1800–1948). Comparison is drawn between U.S. and European practice. Recent German plants for dist of synthetic oils (which are more thermo-stable than natural pet) operate at 2 to 3 mm Hg and up to 425° C. V. B.

1625. Patents. U.S.P. 2,577,701 (20.5.46; 4.12.51). P. H. Deming and M. L. Sagenkahn, assrs to Shell Development Co. Fractionation process for separating mixtures of ethylene and ethane.

U.S.P. 2,577,617 (28.4.47; 4.12.51). J. Hoodig, assr to Shell Development Co. Fractional distillation process for the separation of propylene from ethane in a mixture with methane, propane, and C_{1-4} hydrocarbons in which additional propane is added to the condensate-reflux to increase the propylene in the bottoms liquid product.

U.S.P. 2,577,615 (8.9.47; 4.12.51). A. H. Garrison and F. B. Rolfson, assrs to Shell Development Co. Automatic distillation apparatus.

U.S.P. 2,576,946 (22.6.48; 4.12.51). H. N. La Croix, assr to Foster Wheeler Corp. A bubble cap having a skirt portion normally submerged in liquid on the tray and containing parallel axially elongated vapour passages *A*, has mounted thereon a band of flexible material provided with a corresponding number of vapour-passage-controlling members *B* adapted to close *A* in a fluid-tight manner, the construction being such that vapour flow above a predetermined amount within the cap causes *B* to pivot about a fixed end to an open position against the tension of the flexible material.

U.S.P. 2,580,260 (10.6.50; 25.12.51). E. J. Winters, assr to Lummus Co. Bubble-cap hold-down device comprising a pair of tongs with pivotally connected legs extending downwards through the riser, the out-turned lower end portion of each leg extending over the underside of the deck plate, and a handle projecting through the bubble cap and detachably connected to it so as to hold the bubble cap in operative relation to the riser. V. P. P.

Absorption and Adsorption

1626. " Winterizing " natural-gasoline plant operation. H. W. Wade. *Oil Gas J.*, 10.3.52, **50** (44), 112.—Freezing of water lines, excessive ice formation in water-cooling towers, freeze-ups in " dry " or radiator-type coolers, and freezing of pressure pilot lines and in connexions and insides of cases of liquid-level controllers are main sources of trouble in gasoline plants and compressor stations resulting from cold. These factors are discussed relative to design. G. A. C.

Solvent Extraction and Dewaxing

1627. Liquid–liquid equilibria. J. C. Upchurch and M. van Winkle. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 618–21.—Data are reported on the systems acetic acid–heptadecanol–water at 25° and 50° C, and ethanol–heptadecanol–water at 25° C. E. J. C.

1628. Addition agents and interfacial barriers in liquid–liquid extraction. F. B. West, A. J. Herrman, A. T. Chong, and L. E. K. Thomas. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 625–31.—Experimental work is reported which aimed at investigating the effect of impurities in benzene on the extraction of acetic acid from C_6H_6 drops by water.

Benzenes of various origins and purities were tried in an apparatus using Tygon tubing in the feed system. It was found that the addition of small concentrations of alcohols to the benzenes improved the extraction efficiency of both tall and short spray towers. Results are discussed with reference to the mechanism of mass transfer across films. E. J. C.

1629. Patents. U.S.P. 2,579,867 (19.12.49; 25.12.51). M. L. Sagenkahn, assr to Shell Development Co. In a duosol extraction process the improvement which comprises withdrawing a selected liquid phase *A* from the extraction zone, separating solvent therefrom, and returning remainder of *A* to the extraction zone at a point downstream from the point of withdrawal with respect to the solvent removed, thereby maintaining the ratio of the two solvents different in opposite end zones of the extractor.

U.S.P. 2,580,010 (27.4.48; 25.12.51). M. R. Fenske and B. R. Tegge, assrs to Standard Oil Development Co. Apparatus for liq–liq extraction consisting of a series of superposed stages, each having a mixing zone, a settling zone, and a separating zone adjacent thereto and on the same level, contact between the liquids being effected by means of a gas.

U.S.P. 2,578,685 (10.11.49; 18.12.51). K. A. Fischer. Dewaxing oils by diluting with CH_2ClBr and chilling.

U.S.P. 2,578,510–12 (16.9.47; 11.12.51). E. W. Clarke, assr to Atlantic Refining Co. Waxy mixtures are separated into a higher-m.p. fraction *A* and a lower-m.p. fraction *B* by countercurrent extraction with a solvent *C* and additives *D* so as to form two immiscible liq phases $A + C + D$ and $B + C + D$, from which *C* and *D* are then removed. Per vol of waxy mixture, *C* comprises 1·6 to 2·4 vol $PhNO_2$, D^1 0·25–0·35 anhyd PhOH, and D^2 0·25 to 0·35 vol aq PhOH containing 20 to 30 vol-% water. In 2,578,511, *C* comprises 3·5 to 4·5 vol acetone and *D* 0·3 to 0·5 vol diethylene glycol, *C* being introduced near outlet for *A*, and *D* being introduced adjacent the inlet for the waxy mixture. In 2,578,512, *C* comprises 1·8 to 2·2 vol $PhNO_2$ containing a minor amount of 2 : 2′-dichlorodiethyl ether, D^1 comprises 0·15 to 0·25 vol 2 : 2′-dichlorodiethyl ether, which is introduced adjacent the inlet for the waxy mixture, and D^2 comprises 0·45 to 0·55 vol of a C_{8-18} paraffin introduced between inlet of D^1 and outlet for *B*.

U.S.P. 2,579,501 (14.7.49; 25.12.51). W. W. Kraft, assr to Lummus Co. Separation of wax from a mixture of wax, oil, and solvent chilled with flue gas, in which the flue gas is recovered from the filtrate for re-use in the process together with make-up gas. (U.S.P. 2,580,375, relates to cooling and drying of the combined recovered and make-up gas.) V. P. P.

Cracking

1630. First packaged T.C.C. unit on stream. Anon. *Petrol. Engr*, Mar. 1952, **24** (3), C31.—The 6000-b.d. T.C.C. unit at Artesia, New Mexico, is described with reference to shop fabrication, standardized flow sheets, and prefabrication, which reduce cost. Costs are also reduced by using an oil-well derrick-type structure for the catalytic section, which also serves for rigging the unit. A new liquid phosphoric acid catalytic polymerization unit has also been erected in which a film of catalyst is deposited on quartz by filling a bed of quartz chips with phosphoric acid and then draining.
E. K. J.

1631. Cities Service puts first Orthoflow cat cracker in United States on stream at Ponca City. J. C. Reidel. *Oil Gas J.*, 24.3.52, **50** (46), 200.—The unit, designed by Kellogg Co., has a fresh-feed throughout of 8350 b.d. and operates on the same basic principles as the conventional F.C.C. process. Overall height of 116 ft eliminates some platforms and other structural work.

Process flow, instrumentation, and mechanical design are described; tables show material balance and operating conditions, and a flow sheet is provided. G. A. C.

1632. Orthoflow unit—new feature of refinery. W. T. Ziegenhain. *Petrol. Engr*, Mar. 1952, **24** (3), C26.—The new orthoflow fluid catalytic cracking unit at Ponca City, Oklahoma, handles 8350 b.d. of feed, and includes an internal catalyst stripper to separate catalyst from the cracking products. E. K. J.

1633. Catalytic cracking of high nitrogen charge stock. J. W. Schall and J. C. Dart. *Refiner*, Mar. 1952, **31** (3), 101–3.—Data are presented on the cracking of a 21·2° API, 910° F E.P. gas oil containing 0·4% by wt of nitrogen in a 3-b.d. moving-bed catalytic-cracking pilot plant. At constant cracking temp and recycle ratio yield of gasoline, C_4's, dry gas, and coke increase with an increase in conversion. At constant temp and conversion an increase in recycle ratio increases the gasoline yield and decreases the yield of dry gas, but does not affect the yield of C_4's and coke. Yields of gasoline and C_4's are independent of temp between 900° and 970° F. No significant change in product distribution was found when the catalyst-to-oil ratio was increased from 2·7 to 5·4. Octane ratings of the products are presented. A. R. H.

1634. Catalytic cracking of gas oil. H. Gault and B. Blouri. *Chim. et Ind.*, 1952, **67**, 413–14.—Illustrated description of small laboratory cracking apparatus in which operating temp (700° to 800° C) is attained by combustion of feed stock, excess of latter being fed to cracking section. Results of runs using activated Al_2O_3, brick, and kaolin as cat are given. Only the alumina was free from deposited C at the end of the tests. V. B.

1635. Propane deasphalted gas oil as catalytic cracking feed stock. E. C. Oden and T. S. Granberry. *Industr. Engng Chem.*, Mar. 1952, **44** (4), 896–902.—Describes operating conditions on a commercial down-flow fluid catalytic cracker operating on a mixture of de-asphalted and virgin gas oils. The commercial data are correlated with previous pilot-plant data, and it is shown that the correlations can be used to predict the effect of changing variables on plant yields. It is suggested that the method is applicable to studies of catalytic cracking in general. E. J. C.

1636. Cogelled chromia–alumina catalyst for naphtha reforming—evaluation of hydrocarbon types and naphthas. E. C. Hughes, H. M. Stine, H. A. Strecker, S. C. Eastwood, C. L. Gutzeit, W. A. Stover, and S. J. Wantuik. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 572–5.—Experiments are reported using a chromia–alumina catalyst for reforming naphthas of different hydrocarbon type. Yields and compositions by type are tabulated for a number of different naphthas. E. J. C.

1637. Continuous contact coking of heavy charge material. E. R. Smoley and A. H. Schutte. *Petrol. Process.*, Mar. 1952, **7** (3), 348–9. (*Athabasca Oil Sands Conf., Edmonton, Alberta*, Sept. 12, 1951.)—A continuous-contact coking plant suitable for treating very heavy stocks, such as bitumen from the Athabaska oil sands, is briefly

described. A synthetic crude of low carbon and mineral content, suitable for further refining by conventional means, is produced. Graphical yield data are given for a 4·4° API gravity asphalt of 14 pen and 150° F ring-and-ball m.p. obtained on a semi-commercial unit. The charge meets a stream of hot coke particles of ½ to 1 inch in size, which act as a carrier through the reactor. The particles increase in size on passing through the reactor, from which they are sent to be screened, the selected size being passed to the coke reheater and then on to meet more charge. D. W. F.

1638. Conditions for avoiding the formation of carbon black in the reforming of methane. G. Bellini. *Riv. Comb.*, 1951, **5**, 694–9.—The reactions which can occur in the reforming of methane are considered, and their equilibrium constants at different temps determined.

Through equilibrium constants and conditions of temp and pressure (supposing carbon to be in the gaseous form at the end of the reaction) minimum steam/methane ratio for avoiding carbon-black formation is calculated. (Author's abstract.)

1639. Patents. U.S.P. 2,580,002 (24.12.49; 25.12.51). E. W. Carrier, assr to Standard Oil Development Co. Production of ethylene by thermally cracking a mixture of ethane, propene, and a major portion of propane under ⩽ 2 atm pressure, and discharging the product at 1 atm pressure to a soaking zone containing a Group VI metal hydrogenation catalyst, where concn of ethylene is increased by a reaction involving disappearance of hydrogen and ethane and corresponding formation of methane.

U.S.P. 2,578,377 (24.6.47; 11.12.51). R. P. Smith, assr to Sinclair Refining Co. Catalytic hydrocarbon conversion process in which the finely divided catalyst is subjected to sonic vibrations.

U.S.P. 2,578,704 (8.7.47; 18.12.51). E. J. Houdry, assr to Houdry Process Corp. Reforming of hydrocarbons with dehydrogenation catalysts in a plurality of stages under reaction conditions of successively increasing severity.

U.S.P. 2,579,576 (17.8.49; 25.12.51). J. J. Hickey, assr to Great Lakes Carbon Corp. Cracking with bentonite clay catalyst activated by acid treatment.

U.S.P. 2,576,653 (27.5.48; 27.11.51). C. L. Thomas, assr to Great Lakes Carbon Corp. Production of a cracking cat of long life by cracking hydrocarbon vapours in contact with a raw or acid-treated lattice-type clay to deposit carbon thereon, treating the clay with Cl_2 or a carbon chloride at 300° to 700° C to remove iron, burning off the carbon, and where raw clay has been used, treating with acid.

U.S.P. 2,579,834 (21.5.49; 25.12.51). R. Kollgaard, assr to Houdry Process Corp. Regenerating granular catalysts by passing the catalyst downwardly, as laterally confined compact beds through vertically spaced consecutive combustion zones, the regeneration gas being supplied separately to each zone at progressively decreasing pressures from the lowest to the highest zone, the lowest zone being of greater lateral cross-section than the highest zone, and the flue gas being separately withdrawn from each zone.

U.S.P. 2,579,123 (22.9.48; 18.12.51). W. A. Pardee, assr to Gulf Research & Development Co. Permanently deactivated silica–alumina cracking catalysts are reactivated by impregnating with a fluid compound yielding silica on decomposition and then decomposing it. (In U.S.P. 2,579,133, reactivation is effected with a compound yielding boria on thermal decomposition.)

U.S.P. 2,580,004 (6.2.50; 25.12.51). H. G. Corneil, assr to Standard Oil Development Co. Removal of iron from regenerated, cracking catalyst fines by contacting at 400° to 700° F with HF and passing the mixture into the regeneration zone.

V. P. P.

Hydrogenation

1640. Residuum hydrogenolysis v. coal hydrogenation. A. Clark and W. C. Lanning. *Oil Gas J.*, 10.3.52, **50** (44), 90.—A report on pilot-plant results and preliminary economic studies for residuum hydrogenolysis is given by Phillips Petroleum Co. Advantages over synthetic-fuel production are that raw material is already being

produced as part of crude production, the process dovetails with present refining operations, and hydrogenolysis utilizes many of the techniques employed in production of synthetic fuels with far less drastic transition.

Tables show manufacturing cost of residuum hydrogenolysis, operating data, crude savings by the process, increased production of overhead stocks, and increased supply of naphtha and distillates by hydrogenolysis of 1951 estimated consumption of residual fuel oil. G. A. C.

1641. Cost of hydrogenating coal to gasoline. Industry's versus Bureau's figures. Anon. *Petrol. Process.*, Mar. 1952, **7** (3), 321–3.—The four major points of disagreement between the cost estimates for the establishment of a coal-hydrogenation industry, worked out by the National Petroleum Council and the U.S. Bureau of Mines, are revealed in an interim report of the NPC, presented on January 29, 1952. The NPC estimated capital investment, operating, and maintenance costs much greater. They also allow much more on interest, taxes, and return on investment. The Bureau's figures rely on half the total revenue being derived from chemical by-products. These differences result in NPC estimating gasoline from coal hydrogenation to cost 41c gal compared with Bureau's 12 c/gal. D. W. F.

1642. Technique of carbon monoxide hydrogenation. H. Tramm. *Erdöl u. Kohle*, 1952, **5**, 10–17.—Recent progress in CO-hydrogenation syntheses is given. Nine different cat reaction chambers are described and sketched, covering fixed-bed, liquid-phase, and fluidized-bed types. Newly developed Ruhrchemie/Lurgi fixed-bed chamber is fully discussed. It is stated that using new granulated Fe cat instead of former Co cat, output per unit was increased from 2 to 50 tons/day; cat activity increased six times with equal initial cost and life of cat. Most important technical problem of CO hydrogenation is removal of excess heat formed during reaction in order to keep reaction temp at optimum synthesis value (170° to 320° C, depending on process, equipment, and cat employed). With new chamber, required cooling surface was reduced from 3000 to 235 m^2, reaction space from 14 to 2·5 m^3, and consequent steel requirement reduced from 65 to 9·5 tons, all per 1000 m^3 of reaction gases. Further advantages of new process are far greater scale of operations possible, ease of handling feeds containing varying H_2–CO compositions, much lower undesirable formation of methane, and higher yields of primary products containing more than two C atoms per molecule. It is claimed that experimentally verified formulæ have been deduced for predicting max cooling surface required or max cat chamber dia for given process and reaction conditions. Work done on influence of cat activity, reaction kinetics, nature, and yields of main and by-products is fully described. Economics and versatility of process are reviewed. Process is capable of producing hydrocarbons ranging from gaseous fuels and aviation gasoline to slab and hard waxes; wide range of organic oxygen-containing compounds can also be made, all of which can be most significant commercially. Twelve references. P. V.

Polymerization

1643. Patents. U.S.P. 2,579,095 (10.11.47; 18.12.51). C. S. Sachs and J. Bond, assrs to Alexander H. Kerr & Co. Photopolymerization process which is effected in presence of 0·01 to 5% of a compound $R.SO_2X$, wherein X is halogen and R is aryl, alkyl, or halogen.

U.S.P. 2,579,442 (14.12.50; 18.12.51). H. F. Park and H. A. Walter, assrs to Monsanto Chemical Co. Mass polymerization of ethylenically unsaturated compounds with cyanuric triazide as catalyst.

U.S.P. 2,580,277 (22.6.48; 25.12.51). T. Boyd and F. J. Lucht, assrs to Monsanto Chemical Co. Production of fine-grained polymers by polymerizing vinyl chloride dispersed in an aq medium with an aliphatic acid ester of a polyethylene glycol, the glycol residue having an average mol. wt. of 400 to 4000, and the acid residue having 10 to 20 C atoms.

U.S.P. 2,580,325 (5.8.49; 25.12.51). M. J. Scott, E. F. Jackson, and C. H. Basdekis, assrs to Monsanto Chemical Co. Emulsifying agents and catalyst residues are removed from aq emulsions of vinylidene polymer by treating successively with cation and anion exchange resins.

U.S.P. 2,580,019 (9.10.46; 25.12.51). L. R. Gould and G. G. Wanless, assr to Standard Oil Development Co. Reactors used for co-polymerization of *iso*butene and a polyolefin are cleaned with liq *iso*butylene followed by solvent naphtha.

U.S.P. 2,577,390 (Can. 28.4.48; 4.12.51). W. H. Watson, assr to Polymer Corp. Ltd. Emulsion polymerization of butadiene is terminated by adding lignin.

U.S.P. 2,577,677 (24.12.46; 4.12.51). W. W. Crouch, assr to Phillips Petroleum Co. Polymerization of 1 : 3-butadiene mixed with a minor proportion of styrene in benzene soln in presence of powdered sodium as catalyst.

U.S.P. 2,578,910 (1.6.48; 18.12.51). C. A. Uraneck, assr to Phillips Petroleum Co. Production of synthetic rubber by copolymerizing 1 : 3-butadiene and a minor amount of styrene at $-30°$ to $20°$ C in presence of phenyl (dimethyl) hydroperoxymethane and of ferrous 2 : 2′-dipyridyl sulphate as activator.

U.S.P. 2,580,315 (5.4.47; 25.12.51). H. F. Park, assr to Monsanto Chemical Co. Emulsion polymerization of styrene in presence of a primary C_{8-22} alkylamine ester of a C_{1-5} aliphatic acid as dispersant and a water-soluble polymerization cat having an oxidn-reduction potential of $< -1{\cdot}5$.

U.S.P. 2,577,414 (29.12.48; 4.12.51). G. L. Fraser and H. F. Park, assrs to Monsanto Chemical Co. Production of transparent castings of copolymers of styrene and C_{2-5} glycol maleate by incorporating 0·01 to 1·0%, based on total wt of mixture, of sodium dodecylbenzene sulphonate prior to polymerization.

U.S.P. 2,579,596 (14.7.48; 25.12.51). H. F. Minter and M. M. Leven, assrs to Westinghouse Electric Corp. Production of homogeneous solids by copolymerizing a large volume of a solution of an unsaturated polyester of $\leqslant 1$ dicarboxylic acid and a monomer containing the group $CH_2 = C<$ by dissolving a peroxide catalyst in the solution contained in an open container of circular cross-section, covering the solution with a layer of 1 : 5-pentanediol, and effecting polymerization in a series of stages between 5° to 18° C up to 120° to 150° C, the temp and time of reaction being correlated with the dia of the container. V. P. P.

Alkylation

1644. Alkylation contactor. Anon. *Chem. Engng*, Feb. 1952, **59** (2), 208.—The Stratco contactor has been modified by substituting plain tubes for finned tubes and by mounting horizontally. The H.T.C. is increased from 25 to 90, and the circulator drive is more convenient. More viscous materials can be used owing to decreased drag.

Its use is extended to alkylation of cresols, polymerization of *iso*butylene with $AlCl_3$ catalyst, and to grease manufacture. D. H.

Isomerization

1645. Patent. U.S.P. 2,579,699 (6.12.48; 25.12.51). J. C. Hillyer and J. F. Wilson, assrs to Phillips Petroleum Co. Shift of the triple bond in acetylenic hydrocarbons to a more central position of the chain is effected by contacting a mixture of the hydrocarbon and HF with an Al_2O_3 and/or AlF_3 catalyst at 300° to 700° F. V. P. P.

Chemical and Physical Refining

1646. Union's cobalt molybdate process for refining high sulphur stocks. C. Berg. *Petrol. Process.*, Feb. 1952, **7** (2), 186–9. (*Oil Sands Project Conf., Edmonton, Alberta*, Sept. 12, 1951.)—A new process is described for the treatment of low-grade crudes such as are derived from shale oil and the Athabascan oil sands. Hydrogenation of the sulphur, nitrogen, oxygen, and olefinic compounds is effected by contacting the feed stock with a cobalt molybdate catalyst in the presence of recycle hydrogen. The crude is prepared by coking to give a light and heavy distillate. The process can be used to produce high-octane gasoline by reforming the light coker distillate or jet and diesel fuels by submitting the full-range distillate to a mild hydrogenation treatment. Line

diagrams are given of the two plants together with one of a pilot plant. A summary of processing conditions is given for a Santa Maria full-range coker distillate treated in the pilot plant. Inspection data are presented for low-grade crudes and low-grade coker distillates suitable for refining by this process, together with data on the refined products. An 82% yield of high-grade fuel is obtained with almost complete elimination of nitrogen and sulphur. The application of the catalyst to the Hyperforming process is mentioned. D. W. F.

1647. Desulphurization makes line-gas in West Texas. A. L. Foster. *Petrol. Engr*, Mar. 1952, **24** (3), C13–18.—The desulphurization unit at Bronte, Texas, is designed to handle 10 million cu. ft/day with automatic control which includes seven shut-down switches. The gas is sweetened by countercurrent contact with a regenerated solution of 70% diethylene glycol, 20% mono-ethanolamine, and 10% water, and an amine consumption of *ca* 2 lb/million cu. ft. of gas processed is indicated by initial operating results. About 8000 cu. ft/day of sulphide gas are burnt in a flare with 15,000 to 20,000 cu. ft/day of natural gas. E. K. J.

1648. Hydrogen fluoride extraction of high-sulphur virgin petroleum stocks. A. P. Lien and B. L. Evering. *Industr. Engng Chem.*, Apr. 1952, **44** (4), 874–9.—Desulphurizations of naphtha, kerosine, light gas oil, and crude oil were carried out in a carbon-steel bomb using anhydrous HF. Results, tabulated and plotted, indicate the effect of contact time and settling time, volume % of HF used, temp, and water on the efficiency of the process.

In the case of gasoline HF effected essentially complete removal of S without appreciably reducing aromatics, and at the same time increasing the lead response. Other fractions gave similar improvement in the sulphur content, and in the case of catalytic cracking stocks studies showed improved conversions of the treated against untreated stock cracked under the same conditions. E. J. C.

1649. Patents. U.S.P. 2,576,535 (10.2.48; 27.11.51). W. A. Proell, assr to Standard Oil Co. Separation of *sec*-olefins from admixtures with ethylene by selective esterification with a $C_{>5}$ alkane sulphonic acid of $>80\%$ conc at 40° to 140° F for a contact time of 10 to 60 min.

U.S.P. 2,578,602 (22.5.48; 11.12.51). L. Rosenstein, assr to Texaco Development Corp. Dethiolizing hydrocarbons by extraction with an anhydrous alkanolamine solution of alkali-metal hydroxide.

U.S.P. 2,577,824 (11.8.48; 11.12.51). H. M. Stine, assr to Standard Oil Co. (Ohio). Removing mercaptans from hydrocarbons with charcoal impregnated with aq soln of a strong alkali.

U.S.P. 2,577,823 (6.2.48; 11.12.51). H. M. Stine, assr to Standard Oil Co. (Ohio). Desulphurizing heavy hydrocarbon oil containing 1·0 to 6·5 wt-% S as organic S, by treatment with H_2 in partial pressure of 100 to 500 p.s.i.g. at 650° to 850° F in presence of a contact mass consisting of 50 to 85 mol % Al_2O_3 and <2 and >30 mol-% each of oxides of Cr and Mo, the MoO being pptd on preformed Al_2O_3 at pH 6 to 8. V. P. P.

Special Processes

1650. Sulphur from industrial gases. P. Zurcher. *Petrol. Process.*, Mar. 1952, **7** (3), 333–8.—The subject of sulphur recovery from industrial gases is reviewed from the H_2S removal stage to the plant for conversion to sulphur. For natural gas and others with low H_2S content the iron oxide process seems to be favoured. In this process and the continuous-regeneration development of it, also in the Thylox process, the absorbent is regenerated by air blowing, liberating pure sulphur. In the other processes mentioned the Seaboard, using soda-ash absorbent, the Phenolate, using caustic soda plus phenol, the Shell phosphate, Girbotol, and Alkacid processes, the H_2S is removed during regeneration without conversion.

Three types of modern commercial plants are described with flow diagrams for the conversion of H_2S to sulphur. The Mathieson process operated in the McKamie

(Arkansas) field and at Magnolia, Arkansas, together with the plant operated by Stanolind Oil & Gas Co. in the Elk Basin (Wyoming) field utilize the Claus method of oxidation of H_2S to sulphur. Rather careful reaction control is required. The plant of Hancock Chemical Co. utilizes a German development of the Claus method in which one-third of the H_2S is oxidized to SO_2 with great evolution of heat, which is required to bring up to temp the weak gas mixture processed in this plant. The SO_2 formed meets the remainder of the H_2S with more air before passing to the catalytic reactor. A new process is mentioned which may be able to treat sour hydrocarbon-gas streams without first separating the H_2S. Two methods for converting SO_2 obtained from acid sludge or smelting operations are briefly mentioned. Thirty-five references. D. W. F.

1651. How much it will cost you to recover sulphur from H_2S. J. L. Parker. *Petrol. Process.*, Mar. 1952, **7** (3), 338–9 (from paper *Reg. Mtg. Western Petrol. Ref. Assn., Beaumont, Texas*, Feb. 8); *Oil Gas J.*, 31.3.52, **50** (47), 84.—The merits are discussed of conversion of H_2S to sulphur or direct conversion to sulphuric acid. The latter course is more costly. The economics of sulphur-recovery plants are then considered. If a Girbotol plant is to be employed purely for the purpose of extracting H_2S for conversion to sulphur, then the min size of the sulphur-recovery plant is 50 tons/day, owing to the high cost of the Girbotol plant. If, however, the Girbotol plant can be charged against the attainment of some petroleum product spec, or corrosion prevention, a 20-ton/day plant is feasible. The costs of three smaller plants, 15 tons/day, 10 tons/day, and 5 tons/day, are discussed in some detail. On a conservative basis the 5-tons/day plant is the min considerable. D. W. F.

1652. New 30 ton sulphur recovery plant at Calgary. H. C. Plummer. *Petrol. Engr*, Mar. 1952, **24** (3), C33–6.—Between 30 (summer) and 100 (winter) million cu. ft/day of sour gas will be processed, depending on season, by controlled burning with air to give up to 30 tons/day of elemental sulphur of min purity of 99·5%. E. K. J.

1653. Processing sulphur-bearing gases. C. L. Blohm. *Oil Gas J.*, 28.4.52, **50** (51), 89.—Supply problems, processing steps and economic factors associated with recovery of sulphur from gaseous streams are critically reviewed. A number of references are given. G. A. C.

1654. Synthesis of hydrocarbons and compounds containing oxygen from water and carbon monoxide. H. Kolbel and F. Engelhardt. *Brennstoff-Chem.*, 1952, **33** (1–2), 13–21.—(See Abs. No. 1884, 1951.) *Mtg German Soc. for Mineral Oil Research and Coal Chemistry, Munich*, Oct. 1951.)—Transformation of CO and H_2O in one step, with good yields of liq and solid hydrocarbons and O-containing products. Thermodynamic comparisons are made with conventional Fischer–Tropsch reactions. Hydrocarbons are formed according to the overall equation $H_2O + 3CO = (CH_2) + 2CO_2$, alcohols, viz., ethanol, according to the overall equation $3H_2O + 6CO = C_2H_5.OH + 4CO_2$. These proportions are essential; excess CO causes C deposition on cat, excess H_2O cat deterioration. Only with the H_2O/CO ratio 1 to 3 are max yield of hydrocarbons C_nH_{n2}, 208 g/m³ CO (at N.T.P.) with max cat eff and life expected. All tech gases containing CO and free from cat poisons, S, resins, etc., may be used in place of pure CO. For practically pure CO and gas containing only 29% CO identical yields of 97% were obtained. Such cat can yield 195 g hydrocarbon per m³ CO (at N.T.P.). CO transformation at const space velocity and temp is not influenced by pressure. The O_2 cont of product, however, rises between 30 and 100 atm from 1 to 7·1% corresponding to an alcohol cont from 3 to *ca* 30%. The acid No. rises from 5·3 to 30·5, and the yield of total products from 163 to 216 g/m³ CO (at N.T.P.). The optimum synthesis temp lies between 220° and 230° C, in this region with satisfactory cat life a transformation of 95 to 98% CO is attained, whilst methane formed is only 2·5 or 6% of total hydrocarbons. At temp over 300° C practically only methane is formed. Cat of max activity permitting operation at lowest possible temp is required. Max selectivity for desirable reactions and suppression of secondary reactions is required, the latter being obtained by poisoning cat against these reactions. Metals of group 8 of the periodic system which have special affinity for the very reactive CO are suitable. R. T.

1654a. Continuous molding of petroleum waxes. B. Keyes. *Oil Gas J.*, 14.4.52, **50** (49), 128.—Equipment, similar to that used for years for processing chocolate cakes, has been installed at Beaumont Refinery of Magnolia Petroleum Co. Units comprise a precooler, volumetric depositor, multi-tier cooling conveyor, demoulder, and unloader. Finished slabs are of uniform wt. and shape, have square edges and the desired appearance, and are neither porous nor warped. Thirteen 120-mould frame pressers dealing with 12,500 lb/hr require floor space of 1560 sq. ft. G. A. C.

1655. Manufacture of hydrogen from natural gas. M. Patry. *Chim. et Ind.*, 1952, **67**, 399–409.—Available methods are critically reviewed. Thermal dissociation (at $>800°$ C) is used by some U.S. plants, this process tends to be auto-catalysed by C formed. Main methods employ reaction of CH_4 with either steam or O_2. Processes using steam may either be intermittent (Kuhlmann, no cat, operating at $>1200°$ C; modified water-gas process in which natural gas is also injected into coke bed at 1200° C, the coke having a certain cat activity; Karzhavin process using Ni cat) or continuous in which reaction occurs in special Cr/Ni steel tubes at $>800°$ C. Details (with flow sheet) are given of plant using St Marcet natural gas; as resultant H_2 is required for NH_3 synthesis, CH_4 content must be $>$ about 0·2%, and post-combustion chamber is used to ensure this. Processes described using O_2 conversion of CH_4 are the I.G. (Ni cat, operating at 1200° C, suffers from disadvantage of considerable formation of free C and is limited to gases containing max 30% CH_4) and that developed by the Office National Industriel de l'Azote (O.N.I.A.) operating at the Pierrefitte factory using St Marcet gas. This can use pure CH_4 with no C formation, operating temp is 800° C, Ni-base cat, post-combustion chamber is incorporated to reduce CH_4 content of H_2 to above-mentioned acceptable limit of 0·2% for NH_3 production. O.N.I.A. produces 300 tons/day of NH_3, requiring *ca* 700,000 m^3 H_2, of which 65% is now obtained from natural gas. Together with other NH_3 plants half of present St Marcet gas output of 1 million m^3/day could be used as source of H_2 (*cf* Abs. 1675—1950). V. B.

1656. An American Fischer–Tropsch plant. M. L. Kastens, L. L. Hirst, and R. C. Dressler. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 450–66.—A new type of gas-synthesis process is described which has been developed to the pilot-plant stage by the U.S. Bureau of Mines. It consists of two main stages, gasification of the coal, followed by synthesis of the liquid fuel. Two types of gasifier are described briefly with reference to diagrams, the vertical type being chosen on the basis of experience.

In the gasification stage powdered coal is fed into the unit by three feeder lines. Oxygen produced by the Linde–Franke process and steam at 900° to 1000° F are passed in. Details of gas composition leaving the gasifier prior to cleaning are given. After cleaning, the gas is purified by a diethanolamine scrubber to remove sulphur compounds, which would poison the catalyst in the synthesis stage.

Synthesis takes place at 450° to 525° F with the aid of a reduced iron catalyst at 300 to 350 p.s.i. A "jiggling bed" synthesis reactor is used which is claimed to be more efficient than the fluid type, although it is suggested that the slurry type of process would be even more efficient.

Details are included of separating the synthesized liquid into fractions, data given on the auxiliary equipment, general performance, together with general information on equipment, etc. Forty-six references are appended, together with thirty-nine additional ones referring to the equipment. E. J. C.

1657. OXO Process is developed for Texas Eastman's plant. Anon. *Refiner*, Mar. 1952, **31** (3), 149–50.—A process has been developed for combining carbon monoxide and hydrogen with propylene under high pressure to form butyraldehydes along the general principles of the German OXO process. The aldehydes are then converted to alcohol and acids by the conventional processes. A. R. H.

1658. Oil black. W. H. Shearon, Jr., R. A. Reinke, and T. A. Ruble. *Industr. Engng Chem.*, Apr. 1952, **44** (4), 685–94.—A plant is described for manufacture of carbon black at Continental Oil Black Co.'s Westlake, La., plant. The description includes a flow sheet, operating data, and equipment summary. E. J. C.

1659. Patents. U.S.P. 2,577,788 (26.7.49; 11.12.51). J. H. McAteer and C. E. Morrell, assrs to Standard Oil Development Co. Concurrent dealkylation of aromatic hydrocarbons and dehydrogenation of naphthenic hydrocarbons by reacting an alicyclic hydrocarbon with an alkylated aromatic compound in vapour phase at 500° to 600° C in presence of an activated C catalyst with liquid-feed-to-catalyst ratio of 0·1 to 10 v/v/hr.

U.S.P. 2,576,909 (23.5.50; 4.12.51). J. W. Adams, assr to United States Rubber Co. Coagulating a butadiene polymer synthetic-rubber latex containing carbon black and an alkali-metal sulphonate of <8 C atoms by adding a cationic surface-active agent of 10 to 20 C atoms and a water-sol acid and/or polyvalent metal salt or an acid and an alkali salt.

U.S.P. 2,577,053 (30.4.49; 4.12.51). J. C. Walker, assr to Cities Service Oil Co. Production of oxygenated products by passing normally gaseous hydrocarbons above methane through a series of reactors at >1 atm pressure, mixing oxygen with the feed to each reactor, and maintaining a temp favouring partial oxidn.

U.S.P. 2,577,156 (15.6.48; 4.12.51). N. C. Robertson, assr to Celanese Corp. of America. Mixtures obtained by vapour-phase oxidn of hydrocarbons, containing methyl ethyl ketone *A*, tetrahydrofuran *B*, 2-methyl-tetrahydrofuran *C*, formals *D*, acetals *E*, and cyclic oxides *F* in solution in pentane *G*, are separated by distilling off *G*, removing from the residue a fraction boiling up to 75° C containing *A*, *B*, *C*, *D*, and reduced proportions of *E* and *F*, and heating it with methyl alcohol in presence of a catalyst, separating methylal and acetal conversion products and then *A* and *B* from the residue.

U.S.P. 2,578,475 (9.5.49; 11.12.51). J. H. Hirsch and M. J. Boegel, assrs to Gulf Research & Development Co. Production of H_2 and CO in a spark-ignition I.C. engine by introducing into the cylinder a preformed charge mixture of O_2 and hydrocarbon fuel in which H : C is 3·5 : 1 to 4 : 1, and oxygen : fuel is in mol ratio 0·75 : 1 to 0·90 : 1, and compressing and igniting it.

U.S.P. 2,580,068 (29.4.47; 25.12.51). J. S. Beekley and W. E. Vail, assrs to E.I. du Pont de Nemours & Co. Removing S compounds and CO from a H_2-rich gas free from oxygen by contacting at 200° to 400° C/100 to 500 p.s.i.g. with a Cu–Zn catalyst and then with a methanation catalyst under similar conditions.

U.S.P. 2,580,116 (27.12.48; 25.12.51). H. G. McGrath and L. C. Rubin, assr to M.W. Kellogg Co. Hydrocarbon synthesis process employing a total feed mixture containing 8 to 17 mol-% CO, 8 to 30 mol-% CO_2, and a sufficiently high CO concn of $<50\%$ per pass and H_2 : CO mol ratio $<2·5$: 1 to minimize CO_2 formation and tendency of iron catalyst to de-aerate and agglomerate, gaseous components of the reaction effluent being recycled in amount sufficient to maintain the above composition.

U.S.P. 2,580,039 (16.9.48; 25.12.51). R. C. Morbeck, assr to Standard Oil Development Co. Hydrocarbon synthesis using a dense turbulent mass of finely divided reduced fresh red iron oxide particles sintered after reduction in a hydrogen atm at 1550° to 1650° F and promoted with an alkali-metal carbonate.

U.S.P. 2,577,017 (23.11.48; 4.12.51). L. C. Kemp, assr to Texas Co. Production of deuterocarbon fractions by Fischer–Tropsch process using D_2O.

U.S.P. 2,577,208 (Ger. 1.10.48; 4.12.51). W. Reppe and A. Magin. Production of ketones by reacting an olefin with CO and H_2O in presence of a cyanide of a metal capable of forming a metal carbonyl at 100° to 250° C/<10 atm.

U.S.P. 2,578,144 (15.4.49; 11.12.51). W. A. McMillan, assr to Texas Co. Reacting olefins with CO and H_2 in presence of a metallic Co catalyst, and adding a Co salt of acetyl acetone during the reaction.

U.S.P. 2,580,070 (11.3.48; 25.12.51). R. E. Brooks and I. D. Webb, assrs to E.I. du Pont de Nemours & Co. Synthesis of acyl halides RCOX (wherein R = C_{4-7} *tert-*

alkyl; X = Cl, Br) by reacting an alkyl halide RX with CO in presence of BF_3 at $-25°$ to $+200°$ C/>10 atm.

U.S.P. 2,576,113 (22.4.48; 27.12.51). H. J. Hagemeyer, assr to Eastman Kodak Co. Producing aldehydes by reacting an olefin with CO and H_2 at 50° to 250° C/40 to 700 atm in presence of a cobalt or iron catalyst buffered with the sodium salt of a carboxylic acid. V. P. P.

Metering and Control

1660. Refinery instruments—what makes them tick? Pt 8—Pneumatic transmission. C. E. Shannahan. *Petrol. Process.*, Feb. 1952, **7** (2), 198–203.—A typical example of the use of a pneumatic transmitter receiver and transmitter controller is given which is elaborated to the pneumatic set or cascade control, also described with an example. A force-balance cell for transmitting differential pressure and two types of valve positioners are described with diagrams. D. W. F.

1661. Five ways instruments pay. Anon. *Petrol. Process.*, Mar. 1952, **7** (3), 324–5. (*From paper "Managements' yardstick for instrument costs" by A. L. Keeler, R. P. Fay, E. D. Keiper, and S. V. Agnes.*)—Five fundamental economies are shown to be effected by specific instrument applications on a typical crude-oil-distillation unit. These are, labour savings, utility savings, better product quality and yield, maintenance savings, and protection of personnel and equipment. D. W. F.

1662. Correcting for density and viscosity of incompressible fluids in float-type flowmeters. M. R. Shafer, E. F. Flock, H. L. Bovey, and R. B. Van Lone. *Bur. Stand. J. Res., Wash.*, Oct. 1951, **47** (4), 227–38.—Equations are given which have been verified for the specific cases of five flowmeter tubes with eleven liquid hydrocarbons of different physical properties. Application of the proposed method of calibration and correction of the tubes mentioned gave corrected values within ¼% of the true flow values. W. H. C.

1663. Effect of transient pressures on flow metering. E. C. Fitch and H. M. Wyatt. *World Oil*, Jan. 1952, **134** (1), 218.—A mathematical analysis shows that, for pressure surges of the sine wave form produced by a pump or fast closing valve, recorded rate of flow may be in excess of actual value. In the pure case, having the relevant information, the percentage error may be estimated. The accuracy of the metering system may be appreciably improved by the installation of a pressure de-surger. A. J. H.

Products

Chemistry and Physics

1664. Conversion of methyl*cyclo*hexane and of toluene in contact with aluminium chloride. N. I. Shuikin and L. M. Feofanova. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 1952, 137–44.—Reactions were carried out at atm pressure, 80° C, duration 4 to 24 hr, quantity of anhydr $AlCl_3$ 4 to 20% (methyl*cyclo*hexane) and 4 to 10% (toluene). Methyl*cyclo*hexane yielded 2 to 5% of mixed dimethyl*cyclo*pentanes and smaller amounts of dimethyl*cyclo*hexane and *cyclo*hexane. Toluene gave benzene (approx 1%) and about 2% of the three xylenes, *m*-xylene predominating. V. B.

1665. Isomerization of alkanes under hydrogen pressure in the presence of $AlCl_3$. I. Isomerization of *n*-hexane. M. G. Gonikberg, A. E. Gavrilova, and B. A. Kazanskii. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 1952, 157–62.—Tests were made at pressures up to 1650 atm, temp up to 125° C, duration up to 16½ hr, and with $AlCl_3$/*n*-hexane ratio up to 1. In absence of H_2 considerable cracking occurs, but in presence of H_2 isomer yields of 80% can be obtained. Increase of pressure diminishes and increase of temp increases isomerization rate. Addition of C_6H_6 acts as cracking inhibitor and permits lower H_2 pressure. Main product formed is 2 : 2-dimethylbutane, in formation of which 2-methylpentane is an intermediate product; other isomers identified were 2 : 3-dimethylbutane and 3-methylpentane. V. B.

1666. Synthesis and physical properties of *n*-heptane and 2 : 2 : 4-trimethyl pentane. P. Pomerantz. *Bur. Stand. J. Res., Wash.*, Jan. 1952, **48** (1), 76–81.—Large quantities of *n*-heptane and 2 : 2 : 4-trimethylpentane, of high purity, have been prepared by synthetic methods and purified by fractional distillation for use as standards in the certification of primary reference fuels. A portion of the *n*-heptane was further purified to prepare standard samples for the comparison of precision heat-capacity calorimeters. The synthetic methods used are described, and the physical properties of the compounds recorded. W. H. C.

1667. Catalytic hydrocondensation of carbon monoxide with olefins. 7. Effect of carbon monoxide concentration on its hydrocondensation with propylene and *n*-butylene. Ya. T. Eidus, N. D. Zelinskii, K. V. Puzitzskii, and N. I. Ershov. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 1952, 145–51.—(*Cf* Abs. 1068.)—CO concn was varied 0 to 22% for propylene and 0 to 15·8% for *n*-butylene. In both cases hydro-condensation of olefin/hydrogen mixtures did not occur in absence of CO and was at max for CO content in feed gas of 6 to 8%. Reactions occurring at low (6 to 8%) and high (> approx 14%) CO content are quite distinct. Rapid decrease of hydro-condensation reaction at CO 15 to 20% is in contrast to behaviour with ethylene, where hydro-condensation stlll occurs at these concn. V. B.

1668. Catalytic oxidation of ethylene on magnesium chromite. L. Ya. Margolis and O. M. Todes. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 1952, 52–8.—Oxidn was studied under static conditions at low pressure (0·1 to 2 mm Hg). Cat was magnesium chromite containing excess MgO, with addition of sodium silicate, having an area of 70 m^2/g. Cat activity changes due to simultaneous occurrence of two processes, oxidn and adsorption on cat surface. Cat can be regenerated by treatment with O_2; under dynamic oxidn conditions regeneration of cat surface occurs continually and cat retains prolonged activity. Kinetics of oxidn reaction are discussed. V. B.

1669. Catalytic vapour-phase oxidation of some four carbon hydrocarbons. R. H. Breton, S. W. Wan, and B. F. Dodge. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 594–603.—Apparatus, experimental procedure, and results obtained are described for an investigation into the oxidation of *n*-butane, 1-butene, 2-butene, *iso*butylene, and 1 : 3-butadiene in the vapour phase using various silver, silver oxide, and vanadium pentoxide catalysts. The equipment was checked using ethylene which was oxidized over a silver oxide catalyst, and this resulted in 50 to 60% yields of ethylene oxide, depending upon air–ethylene ratio, contact time, and temp employed.

Plotted data illustrate the effect of contact time, bed temp, and air–hydrocarbon ratio on the yields. Experiments were carried out at atmospheric pressure. A mechanism is put forward to account for the product formation on the basis of a peroxide as the intermediate in each case. E. J. C.

1670. Phase equilibria in hydrocarbon systems. H. H. Reamer, B. H. Sage, and W. N. Lacey. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 609–15.—Phase equilibria for the system *n*-butane–water are reported over the range 100° to 460° F and 0 to 10,000 p.s.i. E. J. C.

1671. Equilibrium constants of the heavier hydrocarbons at sub-atmospheric pressure. M. Van Winkle. *Refiner*, Feb. 1952, **31** (2), 111–16.—Vapour–liquid equilibrium constants are presented for dodecane, tetradecane, hexadecene, and octadecene from 10 to 1000 mm Hg abs and 100° to 700° F obtained from experimental data on binary mixtures of these compounds in a Colburn equilibrium still. Within the range investigated the charts presented show deviations of up to 5% from the experimental values due to the characteristics of the individual mixtures. A. R. H.

1672. Nomograph for vapour pressures of butanes and butenes. D. S. Davis. *Chem. Engng*, Feb. 1952, **59** (2), 206.—From −70° to 10° C for *n*-butane, *iso*butane, butene, *iso*butylene, *cis*- and *trans*-2-butene. D. H.

1673. Carbonaceous deposits from hydrocarbon diffusion flames. J. R. Arthur, P. K. Kapur, and D. H. Napier. *Nature*, 1952, **169**, 372.—Coal gas, methane, ethylene, and propane were burned under the following conditions : as normal flames (*a*) at reduced

pressures with cooled targets above, (*b*) at normal pressures with cooled targets in the flame and envelope; as reversed diffusion flames (temp *ca* 350° C less than normal) with cooled targets above. These variations of combustion had large effects on the character and amount of deposit, which was analysed for C and H content, and solubility in CCl_4, acetone, and pyridine. The soluble fractions later precipitated solid material.

Infra-red spectra of the soluble fractions indicate the presence of carbonyl and hydroxyl groups and strong absorption in the regions 700 to 900 and 2800 to 3100 cm^{-1}. In some cases the ultra-violet spectrum was also studied. H. C. E.

1674. Rearrangement of hexamethyl acetone. S. Barton, F. Morton, and C. R. Porter. *Nature*, 1952, **169**, 373.—3 : 3 : 4 : 4-tetramethylpentan-2-one can be obtained from hexamethyl acetone by reaction at room temp in the presence of benzene and sulphuric acid. A reaction mechanism is suggested. H. C. E.

1675. Spontaneous ignition of organic compounds. C. E. Frank and A. U. Blackham. *Industr. Engng Chem.*, Apr. 1952, **44** (4), 862–7.—Apparatus and experimental procedure are described for determining spontaneous ignition temp for alcohols, α-olefins, *n*-paraffins, and ethers up to 20 C-atm in the mol. Further hydrocarbons studied were benzene, toluene, xylenes, three trimethylbenzenes, and several branched-chain paraffins. The results are tabulated and plotted, and showed that there was some correlation between S.I.T. values and anti-knock rating. Effects of metal catalysts and of additives varied with type of ignition. Suggested that the results should be of value in the development of lubricants of reduced flammability. E. J. C.

1676. Physico-chemical methods for the study of high polymers in solution. M. Baccaredda. *Ricerca Scientifica*, 1952, **22**, 211–49.—The great practical importance of determining average mol. wts., mol. wt. distribution, and mol shape of high polymers is first shown. Reference is then made to fundamental principals, experimental installations, and main results of available methods in deep solution : osmometry, viscometry, light diffusion, diffusion velocity, sedimentation velocity and equilibrium, flow birefringence. Reference is also made to empirical methods suitable for investigating the amount of branching in molten polymers, or in their concentrated solutions, by means of ultrasonic velocity or surface-tension measurements. 195 references. (Author's summary.)

1677. Spontaneous oxidation and inhibitors. C. J. Giuliani and R. B. Thompson. *Riv. Comb.*, 1952, **6**, 22–33.—The spontaneous oxidation of hydrocarbons and the factors which play the most important part in this reaction are mentioned, among which are the formation of chains and the mechanism that passes through the intermediate formation of free radicals of the peroxide type.

Mathematical equations are reported which enable the calculation of the oxidation speed in the various cases. Among the products of oxidation of the hydrocarbons of a gasoline (reaction which is catalysed by the presence of several metal irons of the copper type) volatile hydroperoxides as well as polymerized peroxides, non-volatile and heavy, have been found. These compounds are the principal causes of the formation of gums in gasoline and of carbonaceous deposits in engines.

The effect of an oxidation inhibitor U.O.P. is mentioned and its probable way of reaction. (Authors' summary.)

1678. Studies on silica and alumina gels. M. Prettre. *Bull. Ass. franç. Tech. Pétrole*, 1952 (91), 23–41.—Main investigational methods were thermo-gravimetry and X-ray diffraction; dehydration curves (requiring slow prolonged heating) of hydrargillite show breaks corresponding to a new hydrate, $2Al_2O_3.H_2O$, of boehmite structure. Dehydration has to be carried beyond this point for material to acquire cat activity; prolonged heating at 400° to 500° C leads to formation of a fresh, cat active, phase of alumina, which may be either η, ξ, or γ, depending on starting material. Adsorptive and cat properties of alumina are characteristic of solid phases of abnormal chem composition. Silica-gel properties are greatly influenced by pptn conditions, in active material interface is richer in OH groups than remainder of material. Eighteen references. V. B.

1679. Extractive crystallization—a new separation process. Pt 1. K. A. Kobe and W. G. Domask. *Refiner*, Mar. 1952, **31** (3), 106–13.—Urea selectively forms adducts with straight-chain types of compounds, while thiourea selectively forms similar complexes with many branched-chain and certain cyclic compounds. These complexes are crystalline and are readily broken down by heating or by solution in water, the reactant forming an immiscible organic layer in the latter case. This provides a new method of separating mixtures into their respective classes of compounds. The formation and structure of these complexes are discussed. A. R. H.

Analysis and Testing

1680. Gas analysis by a modified Hempel-method (with an explosion pipette). J. H. Förch. *Chem. Wkbld*, 8.3.52, **48** (10), 145–7.—Reduction of the normal time for the analysis of a gas sample with seven components to 1/3 or 1/2. The accuracy of this method is fully comparable with the results of the Orsat or Bunte method.
M. M. J. J. S.

1681. Analysis from gases to waxes by mass-spectrometry. R. A. Brown, F. W. Melpolder, and W. S. Young. *Petrol. Process.*, Feb. 1952, **7** (2), 204–11.—The general use to which Atlantic Refining Co.'s mass-spectrometer has been put is mentioned, with a brief description of methods of analysis. Modifications such as the replacement of greased stopcocks by teflon stopcocks and solenoid valves are described. Diagrams are given of the sample introduction system employed. The varied applications of this instrument include the analysis of petroleum products obtained from refinery processes and of certain chemicals and solvents. Analysis data are given for three gas oils, wax and wax distillates, alkyl benzene blends, and phenols. Twelve references.
D. W. F.

1682. Mass spectra of some organo-lead and organo-mercury compounds. V. H. Dibeler and F. L. Mohler. *Bur. Stand. J. Res., Wash.*, Nov. 1951, **47** (5), 337–42.—Mass spectral data, obtained with a Consolidated 21–102 instrument, are recorded for tetramethyl and T.E.L. and for dimethyl, diethyl, and di-*n*-butyl mercury. Monoisotopic spectra were calculated by using the isotopic abundance derived from each spectrum. A variation in the lead-isotope abundance was observed in the commercially available lead alkyls. The mass spectra of the lead alkyls are qualitatively similar. The principal portion of the total ionization is contributed by the dissociation of one and three alkyl groups. Hydrocarbon ions, and ions resulting from the dissociation of one or more hydrogen atoms, are small. Hydrides and hydrocarbon ions resulting from intramolecular re-arrangement were observed. A preliminary experiment on a leaded gasoline, using a model 21–103 spectrometer, indicated that sensitivity is sufficient to analyse for a few hundredths of a mole per cent of T.E.L. with a precision of about 2% of the value. W. H. C.

1683. Fine structures of mineral oil colour curves on the basis of investigations with the Pulfrich photometer using spectral dispersion (monochromator). R. Koetschau. *Erdöl u. Kohle*, 1952, **5**, 83–90.—Purpose of extinction measurements of viscous oils by the Pulfrich photometer with monochromator was to establish a precise mathematical colorimetry based on typical colour–curve structures in the visible range. Fundamental theories developed by the author since 1933 can lead to application of colour as factor in overall analysis of mineral oils. Colour is of importance as criterion of refining, ageing, and in determining effect of lube oil additives. Special monochromator used was coupled to Pulfrich photometer. This arrangement facilitated light-transmission measurements at wavelength intervals of 100 Å (and sometimes 50 Å). Six mineral oils (E_{50} 10·2 to 33·2°) were examined in detail, a Pennsylvanian bright stock, a Texas distillate, a synthetic lub oil (made from olefins with $AlCl_3$), and the raffinates obtained therefrom by adsorptive refining. Work was carried out in orange to blue range, with slit widths varying from 0·05 to 0·1 mm and spectral purity of 10 Å achieved with point light source or Hg vap lamp. Typical colour curves of the six oils were obtained plotting a log function of the extinction against λ (abscissa). These curves were convex relative to abscissa. Highly aromatic Texas raffinate curves gave remarkably const values for product of the two co-ordinates, as did aged oils and

asphalt, whilst curves for the synthetic lub oil and its clayed filtrate gave const values for the product of extinction function and λ^2 or λ^3 respectively. Hyperbolic types of curves are indicated. Using " wave number " (*i.e.*, $^1/\lambda$) as abscissa, the above curves become respectively straight lines and quadratic or cubic parabolæ. Remaining oils and their clayed raffinates showed analogous colour curves. A hyperbolic-grid graph is shown for comparison of the various curves obtained. Graphical interpretation of ageing processes in lube oils is described; " colour constant " and " colour index " based on various co-ordinates are deduced. Results on twenty commercial oils are given, indicating assessment of extent of refining and base of oil by optical measurements. Filtered Hg vap light is thought to be sufficiently accurate for colour measurements of visc mineral oils, and the recently marketed Zeiss photo-electric photometer and colorimeter are suitable for future requirements of mineral-oil analysis. P. V.

1684. Sensitive method for the detection of traces of water in oil. J. E. C. Stringer. *Nature*, 1952, **169**, 412.—The method depends on the fact that dry air standing over " wet " oil will take up moisture until the partial R.H. is the same as the vol concn of the water in the oil. An apparatus for measuring the partial R.H. is described.

Results are given for two mineral and two vegetable oils containing from 0·1 to 1% water. It is found that the carbide test sometimes gives high results for mineral oils containing additives, due to chemical reaction between the carbide and additive.

H. C. E.

1685. New method for determination of aromatic rings in oils. L. Robert. *Rev. Inst. franç. Pétrole*, 1952, **7**, 55–61.—Expression has been derived which, unlike previous methods, does not require knowledge of mol. wt. For percentage of carbon atoms in aromatic rings ($\%C_A$) $\geqslant 30$; $\%C_A = 1039{\cdot}4\ n_D^{20} - 470{\cdot}4\ d_4^{20} - 0{\cdot}315P - 1094{\cdot}3$, where P is the an. pt. in ° C. Results obtained with this expression are shown to be comparable with those by other procedures. For $\%C_A > 30$ a correction has to be applied, $\%C_A$ (corr.) $= \frac{1}{2}\%C_A + 15$. Anomalous (neg) results can be obtained with certain over-refined and synthetic oils, but procedures involving mol. wt. likewise give anomalous results in such cases. It is emphasized that $\%C_A$ can be very different from content of aromatic hydrocarbons in the oil (which is always greater than $\%C_A$) and that no simple procedure for determination of such aromatic content is available.

V. B.

1686. Sunbury oil test engine. R. Stansfield. *Schweiz. Arch. Ang. Wiss. Tech.*, 1952, **18**, 46–62 (in English).—Single-cyl spark-ignition engine has been designed which can be used to reproduce all more important data obtainable from a variety of lub-oil test engines, either spark or diesel. Design allows for measurement of temp at piston crown, behind piston-rings, at crank-pin, and in cyl liners, these and crankcase temp being crit with respect to rate of oil breakdown. Cyl jacket and head are cooled by independent systems using different coolants if necessary. Control of piston-crown temp can be achieved by jet of lub oil from top of connecting-rod to underside of piston crown. This temp can be reduced to 150° C by keeping head and jacket coolants at 100° C and using oil jet. It can be increased to 280° to 300° C as required for aircraft-engine correlation, without supercharging, by using thick (0·312 inch) cyl liner with dry jacket, ethylene glycol at 184° C as head coolant, and no oil jet to piston crown. Lub-oil temp is controlled by radiant-type heater below sump, and cooler on pump discharge. All lub-oil pipes are made of steel to avoid cat effect of Cu. Blow-by meter is fitted to crankcase, but ring stick is invariably indicated by rise in piston-ring belt temp. Correlation with accepted oil-test engines is obtained, provided following basic principles are observed: (*a*) piston-ring belt temp must be same as in engine being copied; (*b*) temp of oil in sump must be same; (*c*) fuel must contain T.E.L. or sulphur in same concentration as fuel in original test. Piston-ring clearance must be closely controlled. Recommended clearance 0·003 inch, rising to 0·0045 inch if longer test period required. Oil consumption may be regulated by use of taper-faced rings. Metals having cat effect should be introduced if they occur in primary test engine. Tests to ring-stick can be made with high degree of reproducibility, but runs of definite duration are preferred, with a demerit system for rating if no ring-stick occurs. Caterpillar L-1 test is simulated very reliably by running at 2500 r.p.m., 80 b.m.e.p., 60° C oil to bearing, head cooling 100° C, dry jacket, oil pressure adjusted to give 180° C behind top ring groove, duration of test 150 hr.

A. C.

1687. Patent. U.S.P. 2,577,640 (18.11.48; 4.12.51). E. F. Wadley and J. A. Anderson, assrs to Standard Oil Development Co. Analysis of hydrocarbon fractions boiling in the gasoline–gas oil range and containing dienes by blending with a liquid-saturated hydrocarbon, treating part of the blend with a solution of HNO_3–$Hg(NO_3)_2$ or $KMnO_4$ to remove dienes and comparing the absorption of light of 235 and 258$\mu\mu$ of the treated and untreated blends. V. P. P.

Gas

1688. Classification of combustible gases. M. B. Ravich and N. V. Lavrov. *Izvest. Akad. Nauk S.S.S.R., Otdel. Tekh. Nauk*, 1952, 72–80.—Variations in properties of combustible gases are noted, cal. val. can cover 80 : 1 range (300 to 25,000 k. cal/m³), vol air for combustion 100 : 1 (0·3 to 28 m³/m³), vol combustion products 25 : 1, combustion temp 2 : 1 (860° to 2200° C). Classification on basis of combustion temp is proposed, highest val is pure CO (2370° C). Five groups are suggested >2000° C (<25% inerts), 1700 to 1900 (35 to 50%), 1500 to 1700 (50 to 70%), 750 to 1300 (>70%), <750, latter group, *e.g.*, exhaust gases from I.C. engines, is not at present used, and can only be burnt by cat processes. Another suggested classification is based on potential H content ($H_2 + CO + (2n + m/2)\,C_nH_m$, four groups being proposed, >500, 200 to 400, 80 to 200, <80%. Tables are given for both classes showing properties and analyses of industrial gases. V. B.

1689. Hydrates in the natural gas industry. J. P. Lacroix. *Rev. Inst. franç. Pétrole*, 1952, **7**, 34–49; 62–79.—Comprehensive survey, based almost exclusively on published sources. Hydrates can be formed by methane, ethane, propane, butane, and (probably) *iso*butane. Laboratory work is surveyed, and numerous graphs and tables (all reduced to metric units) are quoted. Methods of analysis of hydrates are described and compositions obtained by various workers compared. Temp of hydrate formation can be lowered by inhibitors, of which NH_3 and CH_3OH are most effective. Conditions governing hydrate formation are discussed with some data on St Marcet gas field. Methods of gas dehydration are described. Storage of natural gas in hydrate form (at approx −45° C) is outlined, economics of this process are doubtful. 221 references. V. B.

1690. Acetylene from natural gas. P. Gustavson. *Nafta* (*Yugoslavia*), Nov. 1951, **2** (11), 15–20.—The general importance of acetylene as a raw material for the organic chemical industry is described, and new processes for the manufacture of acetylene from natural gas are discussed (Huls, Sachse, Wulff, and Schoch processes). Finally, the requirements for acetylene in Yugoslavia, and the aspects of the various processes under the present situation in that country are considered with special respect to the availability of raw materials and power.

1691. Higher overall furnace heat-transfer and furnace walls heat absorption by the combustion of natural gas. M. Medici. *Ricerca Scientifica*, 1951, **21**, 1942–52.—An attempt is made to demonstrate that overall furnace heat-transfer rate and furnace-walls heat-absorption are higher by the combustion of natural gas than by the combustion of other fuels. Advantages of the combustion of natural gas claimed are more radiating power of non-luminous gas constituents, better quality-index for the combustion process, more uniform hot-gases temp-distribution in the furnace, and a higher effective heat-tonality of the combustion products. A detailed investigation is made of the various performance-factors, and the result is that natural-gas combustion is in many respects the combustion of an ideal fuel. (Author's summary.)

Engine Fuels

1692. Fuels for use in marine auxiliary oil engines. C. D. Brewer. *Motor Ship*, Mar. 1952, **32** (384), 514; *Trans. Inst. mar. Engrs*, Apr. 1952, **64** (4), 59–76.—Two completely different classes of fuels are considered, diesel fuels, which are divided into two classes under BS 209/1947, and boiler or heavy fuels. Data from a number of tests on four different engines with fuels of various visc show that there is little relationship between fuel density and max brake thermal efficiency. Data are also

given to show the influence of ignition quality of the fuel on engine performance, which is less than might have been expected. Results of experiments show that cetane no. of diesel and heavy fuels could be at least as high as that of some gas oils, and that ignition quality of a fuel is determined essentially by the type of crude from which it is derived. Visc has been shown to have little influence on ignition quality. The results of these experiments may provide part of the explanation of the smoother operation of main engines when running on some heavy fuels than on some diesel fuels. Results of tests of the influence of fuel sulphur content on piston ring and cyl bore wear are given. The extent of deposits built up on the tip of the atomizer was shown to bear a direct relationship with certain fuel characteristics, such as Conradson carbon and sulphur (particularly if both are high), but this deposit is also influenced by operating conditions. Periods of interference to combustion efficiency by nozzle tip deposits resulting in increased exhaust temperature are illustrated by a recorded trace of the temp when operating under conditions favourable to formation of these deposits. Tests were conducted on two engines, a four-stroke and a two-stroke of 240 and 212 mm bore respectively, and showed that correlation between fuel properties and max brake thermal efficiencies was not clearly evident. There was no major difficulty in operating for short periods on heavy fuel oils provided that they were pre-heated to give a visc of no more than 150 sec R.I. at the injection pump inlet. Data show the effect of use of chromium-plated bores on ring and bore wear, and also the reduction of wear with a small supercharged engine operated under conditions of moderate boost.

U. M.

1693. How jet fuel is blended to specifications. B. G. Adams. *Refiner*, Feb. 1952, **31** (2), 95–6.—The following stocks were available for jet-fuel blending in the proportions given, kerosine distillate (500° F.E.P.) 40 to 50%; naphtha (300° to 410° F) 36 to 46%; casinghead (22 lb R.V.P.) 10 to 20%; *n*-butane (56 lb R.V.P.) 0 to 3%; the controlling specifications being R.V.P., gravity, and freezing point. The exact proportion of casinghead was calculated by assuming a linear relation between R.V.P. and % casinghead. If this revealed an amount in excess of that available, *n*-butane was added, the amount being determined experimentally in an apparatus which is described. The max proportion of high-gravity material was determined by the relation

$$\% \text{ high-gravity material} = \frac{H - D}{(H - D) + (D - L)}$$

where H = API gravity of high-gravity material; D = desired API gravity; L = API gravity of low-gravity material. Water precipitates during storage and is of little importance. The freezing point was found to be controlled by the congealing point of the hydrocarbons in the kerosine distillate. The actual freezing point is indicated by the amorphous congealing of hydrocarbons which disappear when the oil is removed from the coolant.

A. R. H.

1694. Nomograph for influence of sulphur compounds on T.E.L. efficiency. G. E. Mapstone. *Refiner*, Feb. 1952, **31** (2), 132.—The loss of T.E.L. efficiency in the presence of thiophenes, free sulphur, sulphides, carbon disulphide, disulphides, mercaptans, and polysulphides is given with an accuracy of about ±5%.

A. R. H.

1695. Patents. U.S.P. 2,580,243 (30.4.51; 25.12.51). E. B. Rifkin and D. H. Ewen, assrs to Ethyl Corp. Gasoline containing T.E.L. is deleaded by treatment with a nitrogen oxide.

U.S.P. 2,576,458 (7.4.50; 27.11.51). E. F. Hill and F. H. Baldwin, assrs to Ethyl Corp. As anti-oxidant, a substituted ethanolamine of general formula

HOCHX.CHY.NHB

wherein one of X and Y is hydrogen and the other is alkyl, phenyl, alkoxymethylene or phenoxymethylene, and B is *p*-amino-, *p*-hydroxy-, *p*-(N-alkylamino)- or 2- or 3-alkyl-4-hydroxy-phenyl.

U.S.P. 2,579,692 (9.12.49; 25.12.51). J. F. Neudeck, assr to Standard Oil Development Co. Gasoline fuel containing *ca* 0·5 vol-% solvent oil and 1·0 to 2·5% of dimethyl carbinol, said solvent oil consisting of a liq hydrocarbon mixture having a kauri-

butanol solvent power above *ca* 20, a 50% dist point >350° F/10 mm, visc >450 S.S.U/100° F, and an API gravity 18° to 28°.

U.S.P. 2,580,015 (31.12.47; 25.12.51). R. S. George, assr to Hercules Powder Co. Diesel fuel containing 0·25 to 5 wt-% of an α, α-dialkylaryl methyl hydroperoxide. V. P. P.

Gas Oil and Fuel Oil

1696. Patent. U.S.P. 2,579,890 (3.7.48; 25.12.51). C. Wies, J. B. R. Caron, and E. B. Glendenning, assrs to Shell Development Co. Burner-screen clogging caused by cracked components in fuel oil is inhibited by incorporating *ca* 0·005% sodium dioctyl sulphosuccinate, *ca* 0·0042% barium salt of alkyl phenol disulphide, and *ca* 0·0265% diethylene glycol monobutyl ether. V. P. P.

Lubricants

1697. Lubrication problems in plain bearings. W. Frössel. *Erdöl. u. Kohle*, 1952, **5**, 167–70.—Lubrication theory of a plain bearing is discussed and illustrated with diagrams. Examples of three methods of oil feed to bearings are depicted. V. B.

1698. Soap structure of lubricating greases revealed by electron microscope. R. R. Matthews. *Petrol. Process.*, Feb. 1952, **7** (2), 181–2.—The salient points are given of four papers presented at the Ann. Meeting Nat. Lub. Grease Institute, Chicago, Oct. 29 to 31, 1951. Three of the papers are concerned with the use of the electron microscope for revealing the fibrous structure of the densifying soaps and the effects of working, etc., on them. They are: "The structure of gels of hydrous calcium stearate in cetane: rheological studies and electron microscopy," by R. D. Vold, H. F. Coffer, and R. F. Baker; "Developments in the field of soda-base lubricating greases," by A. Bondi; "Electron microscope studies of lithium lubricating greases," by J. A. Brown, C. N. Hudson, and L. D. Lorings. Another paper summarized is "Phthalocyanine lubricating greases," by R. L. Merker, C. R. Singleterry, and V. G. Fitzsimmons. D. W. F.

1699. Fundamental aspects of lubrication. Anon. *Nature*, 1952, **169**, 363.—Summary of a conference held by the New York Academy of Sciences, Mar. 1950. The subjects discussed include full-fluid lubrication, thin-film lubrication, influence of surface film on solids, X-ray- and electron-diffraction studies of lubricating surfaces, surface chemical phenomena, adsorbed films in the lubrication of graphite sliding contacts, visc and molecular structure in relation to synthetic lubricants, measurement of incremental friction coefficient, influence of oxidation and roughness on wear of lubricated metal surfaces, fine structure of lubricating grease, and the process of metal cutting. H. C. E.

1700. Friction and lubrication. D. Tabor. *Nature*, 1952, **169**, 393.—Report of a discussion held under the auspices of the Royal Society, London, on Apr. 19, 1951. Papers were presented under the following headings:

(i) *Metallic Friction.*—The basic mechanism of friction was discussed, as well as the frictional props of certain low-friction materials, *e.g.*, porous solids impregnated with polytetrafluorethylene, or Mo_2S_3 chemically formed on Mo surfaces. The effect of surface oxide films in preventing metallic contact and the importance of the Beilby layer were emphasized. Metallic wear involves the formation and shearing of metallic junctions at the regions of real contact.

(ii) *Friction of Non-metals.*—Plastics exhibit deviation from Amonton's Law at low loads, but apart from this their behaviour is the same as that of metals, with the exception of polytetrafluoroethylene, the molecular structure of which endows the material with very low frictional properties.

(iii) *Boundary and Extreme-pressure Lubrication.*—The most effective lubrication is obtained when the lubricant is solid and has a close-packed structure. On melting increases in friction and chemical reaction occur, and at high temps desorption of the lubricant film may occur. An optical method of measuring the thickness of adsorbed

film was described. Extreme-pressure lubricants contain additives (Cl or S compounds) which are inert under ordinary conditions but react with the surface at the high temps of incipient seizure to form protective films. H. C. E.

1701. Phthalocyanine lubricating greases. V. G. Fitzsimmons, R. L. Merker, and C. R. Singleterry. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 556–63.—Details are given of experimental work carried out to investigate the use of phthalocyanine pigments on gelling agents for greases. Greases gelled in this manner were found to have good characteristics at temp above 150° C, and have a tendency to stiffen rather than soften at higher temp. Test results are plotted and tabulated. E. J. C.

1702. Patents. U.S.P. 2,580,053 (30.8.49; 25.12.51). T. S. Tutwiler, J. C. Mundy, and D. T. Rogers, assrs to Standard Oil Development Co. Mineral lub. oil containing 0·01 to 15 wt-% of a copolymer of an ester $CH_2 = CY.COOR$ (R = alkyl; Y = H, Me) with an ester $R'OOC.CY' = CH.(CH_2)_n.COOR''$ (R′, R″ = alkyl having an average of $\geqslant C_8$; Y′ = H, Me; $n = 0, 1$; No. of C atoms in R minus average No. of C atoms in R′ and R″ = 5 to 20), the proportions being such that the copolymer has an average side chain of 4 to 15 C atoms.

U.S.P. 2,580,038 (30.9.49; 25.12.51.) L. A. Mikeska, assr to Standard Oil Development Co. Mineral lub. oil containing a minor proportion of a product obtained by heating a salicylic acid ester, in which the ester group contains 8 to 24 C atoms, at 50° to 250° C for 2 to 20 hr with an equimolar amount of formaldehyde.

U.S.P. 2,577,719 (23.5.50; 4.12.51.) W. T. Stewart, assr to California Research Corp. Mineral lub oil containing 0·1 to 2 wt-% (based on finished oil) of 1 : 1′-dicarboalkoxy-8 : 8′- and/or 9 : 9′- heptadecyl selenide (wherein alkoxy has $\geqslant 8C$).

U.S.P. 2,580,274 (8.3.47; 25.12.51). R. F. Bergstrom and J. M. Plantfeber, assrs to Shell Development Co. Heavy-duty lubricant comprising a mineral lub. oil and minor amounts of: (*a*) an oil-soluble (Ca) salt of a pet sulphonic acid; (*b*) an arylamine (phenyl α-naphthylamine); (*c*) a salt of a methylene bis(alkylphenate), *e.g.*, Ca salt of octyl phenolformaldehyde condensation product; (*d*) a salt of a dialkyl dithiocarbamate (zinc dibutyl dithiocarbamate).

U.S.P. 2,578,689 (27.8.46; 18.12.51). M. W. Freeman. Lubricant with E.P. and corrosion-inhibiting properties containing a product obtained by reacting a water-sol pet sulphonic acid with 10 to 40 wt-% phosphorus sulphide at $\geqslant 450°$ F.

U.S.P. 2,579,777–8 (6.4.49; 25.12.51). W. S. Allen, assr to United States Steel Co. Lubricating strip metal with an oil-in-water emulsion of pH 2 to 6 produced by a weak organic acid, the oil being in (*a*) a glyceride oil (palm, coconut, lard, etc.) or in (*b*) dibutyl phthalate, sebacate, or citrate, with 5 to 25% of a C_{14-18} carboxylic acid, emulsified with a cationic or non-ionic emulsifier.

U.S.P. 2,580,005 (31.12.47; 25.12.51). E. B. Cyphers and G. M. McNulty assrs to Standard Oil Development Co. E.P. lubricant consisting of a mineral lub. oil containing 1 to 20 wt-% of a sulphurized fatty oil in which is dispersed a minor proportion of a phosphorus sulphide and a metal deactivator.

U.S.P. 2,578,851 (12.9.49; 18.12.51). H. G. Smith, T. L. Cantrell, and M. L. Hill, assrs to Gulf Oil Corp. A soap–oil lubricating grease containing a metal phthalyl alkyl amide, in which alkyl contains 8 to 20 C atoms, to prevent bleeding.

U.S.P. 2,577,706 (26.11.48; 4.12.51). B. W. Hotten, assr to California Research Corp. High-melting, high-water-resistant grease consisting of a lub oil, an alkali-metal organic acid salt as thickener, and a polymeric compound containing recurring saponifiable groups (carboxyl or carboxy-ester), of which 10 to 60% are saponified with alkali or alkaline-earth metals. V. P. P.

Bitumen, Asphalt, and Tar

1703. Modern asphalt refinery can be neat and trim. Anon. *Petrol. Process.*, Mar. 1952, **7** (3), 332.—A brief description with photographs is given of the 3500-b.d. asphalt refinery at Richmond Beach, Wash., of Stancal Asphalt and Bitumuls Co. which was put into operation in June 1950. D. W. F.

1704. The chemistry and rheology of asphaltic bitumen. S. L. Neppe. *Refiner*, Feb. 1952, **31** (2), 137–42.—The methods used in the petroleum industry for the production of straight-run, cracked, and air-blown bitumens are outlined, and the literature on its constitution is reviewed. 103 references. A. R. H.

1705. Momentary reactions between dry gases and solutions of asphaltic bitumen and tar. F. J. Nellensteyn and J. E. W. van den Driessen Mazeeuw. *Chem. Wkbld*, 15.3.52, **48** (11), 158–60.—The study of momentary reactions between asphaltic bitumen and dry gases has been extended to various gases (HCl, HBr, HJ, HF, BF_3, SiF_4, $COCl_2$) and bitumens (Gilsonite and bitumens from Venezuela, Mexico, Trinidad). The result is a confirmation of a formerly published hypothesis, that these reactions are due to dissociation products, so-called free radicals (*Chem. Wkbld*, 1942, **39** (29), 394–5; *ibid.*, 1945, **41** (1/2), 5–7). The determination of the magnetic susceptibility over a wide temperature range gives an indication in the same sense. Some applications of these reactions are mentioned. M. M. J. J. S.

1706. Protection of canal and river banks and of sea walls by flexible pre-fabricated bituminous coatings. J. Filippi. *Bull. Ass. franç. Tech. Pétrole*, 1952 (92), 3–26.—Pre-fabricated bituminous coatings can be applied *in situ*, under water if necessary, and shape themselves to support; when repair is necessary this can be easily accomplished. Coatings are 4 to 15 cm thick and reinforced with metal or rope. Compositions quoted are: sand 66 to 80%, filler (200 mesh) 10 to 22%, and bitumen (31 to 70 pen) 12 to 17%. Examples of use of such coatings in the U.S.A., Holland, and France are given, with details of latter. Tests used are discussed (stability, fragility, weathering, frost-resistance, flexibility, thermal conductivity, flow, tensile strength). Manufacturing procedure and methods of transport of finished coatings are described. Numerous illustrations. V. B.

1707. Patents. U.S.P. 2,579,652 (12.1.48; 25.12.51). H. F. Dague, assr to Asphalt Specialities Corp. A readily pumpable asphalt suspension containing 50 to 65 wt-% powdered asphalt, 50 to 35 wt-% water, and 0·5 to 3% of an oil-soluble, anionic surface-active sulphonate.

U.S.P. 2,580,035 (2.12.48; 25.12.51). C. Mack, assr to Standard Oil Development Co. Flow properties of an asphalt not having Newtonian flow properties are improved by incorporating 0·5 to 5% of α-nitronaphthalene. V. P. P.

Special Hydrocarbon Products

1708. Mineral oils for plant protection. B. Waeser. *Erdöl u. Kohle*, 1952, **5**, 129–30.—Review of past and present application of mineral oils as plant-protective agents. Description is given of requirements regarding physical and chem properties of impregnating and preserving agents, insecticides, and emulsifiers made from pet-base oils. P. V.

1709. Petrolatum from Emsland crude. W. Fuchs and W. Lommerzheim. *Erdöl u. Kohle*, 1952, **5**, 148–51.—Petrolatum obtained on pilot-plant scale from Emsland (Emlichheim) crude had d_4^{70} 0·838, m.p. 63·5° C, E_{50} 2·96°, mean mol. wt. 675, S 0·33%, C/H 1/1·89, iodine val 7·5, acid val 0·02. Separation into components was by fractionation cryst from 1 : 2-dichlorethane, further fractionation was by adsorption on active C and silica gel from solution in CS_2; interpretation of results was largely based on relationships derived by Grodde (*cf* Abs. 1123—1950). Conclusions are that mol. wt. of hydrocarbons of petrolatum is 450 to 1000, lowest mol.-wt. fractions being naphthenic oil; hard paraffin components (m.p. *ca* 75° C) have mol. wt. 500 to 700 and do not contain *n*-paraffins. Chain-branching of hydrocarbons increases with mol. wt., at higher values of the latter (*ca* 800) an appreciable proportion of ring components is present. It is thought that petroleum-jelly characteristics are associated with such ring compounds. V. B.

1710. Meeting product quality in wax crystallization. H. A. Lund. *Petrol. Process.*, Mar. 1952, **7** (3), 326–31.—The results of an investigation into the different types and properties of crystals formed from a wax are reported. A special de-oiled narrow-b.p.

wax fraction was separated into fifteen fractions by repeated crystallization. From photomicrographs of several fractions it can be seen that three crystal types are formed—plates, having the highest m.p., needles, with intermediate m.p., and micro, with lowest m.p. Tensile studies show that plate-type waxes have low strength. Needle and micro have high tensile strengths. High-strength waxes can be made by blending different fractions. Wax hardness is directly related to m.p., also to crystal type, and can be varied by blending. A pressibility test is described for determining the ease with which a wax can be de-oiled. Photomicrographs again show that plates give excellent pressibility, and by blending the pressibility of needle- and micro-type waxes can be raised to a reasonable amount. The crystal type of a certain fraction is dependent upon the solvent used for crystallizing out. Plate crystals formed in absence of solvent are not so much affected by the solvent, but micro- and needle crystals may change with different solvents used. D. W. F.

1711. Take another look at coke. Anon. *Petrol. Process.*, Mar. 1952, **7** (3), 351–2.—The demand for petroleum coke is now outstripping production, as the greater quantities of high-sulphur crudes now being processed do not give the low-ash, low-sulphur coke required for metallurgical uses and electrodes for aluminium making. The price of coke may now warrant the running of selected feed stocks, pretreated to reduce sulphur content. D. W. F.

1712. Patents. U.S.P. 2,578,432 (Fr. 27.6.30; 11.12.51). J. P. Labour, assr to Gasoline Research Industrial & Commercial Co. Process of producing apparent solidification of liq hydrocarbons *A* by forming an aqueous emulsion of *A* and an alkali-soluble methyl cellulose (Tylose) and then adding H_2SO_4.

U.S.P. 2,577,840 (22.6.50; 11.12.51). R. R. Burnham, A. T. Anderson, and A. M. Matheson, assrs to Union Carbide and Carbon Corp. Compositions for use in aq baths for waterproofing textiles comprising 35 to 70 parts wax, 1 to 2 parts amine emulsion stabilizer, 12 to 20 parts polyvinyl alcohol emulsifier, 10 to 20 parts water-sol fatty acid salt of Al or Zr, and up to 10 parts buffer containing sodium diacetate.

U.S.P. 2,580,050 (29.12.44; 25.12.51). W. J. Sparks and P. E. Hardy, assrs to Standard Oil Development Co. A self-sustaining film consisting of 2 to 10 wt-% of a microcrystalline pet wax and the remainder of a copolymer of a styrene and *iso*-butylene containing 50 to 60% of combined styrene.

U.S.P. 2,577,643 (11.10.49; 4.12.51). D. W. Young and A. J. Morway, assrs to Standard Oil Development Co. Wax composition consisting essentially of refined paraffin wax and 1 to 5% of an alkali- or alkaline-earth-metal salt of a heterocyclic carboxylic acid of low mol wt having a five-membered furan-type ring.

U.S.P. 2,577,816 (23.5.46; 11.12.51). H. G. Schneider, D. W. Young, and J. P. Rocca, assrs to Standard Oil Development Co. Composition comprising 1 to 50% polyethylene in chlorinated wax containing 2 to 8% Cl.

U.S.P. 2,577,218 (Neth. 19.12.46; 4.12.51). M. van der Waarden, assr to Shell Development Co. Oil concentrate capable of forming a stable, non-gelling water-in-oil emulsion comprising an oil containing 0·5 to 15% of free fatty acid fraction of degras as emulsifier and 0·02 to 5% of colophony and/or acidic fractions of colophony to inhibit gelation.

U.S.P. 2,579,428–31 and 2,579,434 ((*a*), (*b*), (*e*) 2.2.49; (*c*) 11.6.49; (*d*) 9.3.50; 18.12.51). O. H. Hammer, assr to Dow Chemical Co. Compositions for controlling mites comprising 4-chlorophenyl 4-chlorobenzene sulphonate with mutually activating amounts of: (*a*) di(4-chlorophenoxy) methane; (*b*) sulphur; (*c*) triethanolamine salt of 2 : 4-dinitro-6-*sec*-butylphenol; (*d*) acid lead arsenate; (*e*) 4-nitrophenyl diethyl thiophosphate.

U.S.P. 2,579,296–301 (8.4.48; 18.12.51). G. A. Buntin, assr to Hercules Powder Co. Insecticidal compositions comprising chlorinated terpenes derived from: (*a*) pinene; (*b*) camphane; (*c*) bornyl chloride; (*d*) *iso*bornyl chloride; (*e*) pinane; (*f*) fenchene.

U.S.P. 2,578,736 (29.5.46; 18.12.51). M. Pijoan, R. D. Englert, H. J. Gerjovich, and M. L. Hopwood. Insecticidal composition comprising an insect penetrant solvent and a compound consisting of two partially hydrogenated naphthalene rings condensed with a chloral group, the rings containing a OH, OAlk, halogen, or carboxylic acid ester group substituent.

U.S.P. 2,577,969 (13.8.45; 11.12.51). F. D. Jones, assr to American Chemical Paint Co. Herbicide comprising oil-sol pet sulphonates in amount sufficient to dissolve 2 : 4-dichloro- or 2 : 4 : 5-trichloro-phenoxyacetic acids, or their amides, thioamides, esters, and salts.

U.S.P. 2,580,048 (12.4.48; 25.12.51). A. J. Shmidl, assr to Standard Oil Development Co. Fungicidal emulsion comprising a copper salt of a C_{1-4} alkylated phenoxyacetic acid, a wetting agent, a heavy oil, and water.

U.S.P. 2,577,093 (6.11.48; 4.12.51). M. Silverman, assr to Schieffelin & Co. Acylamido naphthoquinones as fungicides.

U.S.P. 2,577,041 (10.4.48; 4.12.51). R. B. Seymour, assr to Industrial Research Institute of the University of Chattanooga. Production of fungicides by condensing a copolymer of maleic anhydride and a monovinyl compound with a chlorophenol or a mono-nitrophenol. V. P. P.

Derived Chemical Products

1713. Patents. U.S.P. 2,577,729 (31.5.50; 11.12.51). W. V. Bauer, assr to Stauffer Chemical Co. Apparatus for CS_2 manufacture comprising an elongated tubular shell standing vertically in a furnace and divided into upper and lower zones by a transverse support. C is fed into the top zone and S into the lower. The lower zone is provided with a tubular insert concentric with the shell which has on its outer periphery adjacent to the shell a spiral fin which provides a spiral passage for more rapid flow of S vapour. In U.S.P. 2,577,786 the lower zone is provided with a tubular vessel having a closed conical top spaced from the adjacent shell.

U.S.P. 2,578,720 (22.12.45; 18.12.51). E. T. McBee and R. M. Robb, assrs to Purdue Research Foundation. Fluorination of fused-ring compounds with CoF_3.

U.S.P. 2,578,721 (29.12.45; 18.12.51). E. T. McBee and R. M. Robb, assrs to Purdue Research Foundation. Fluorination of saturated halocarbons with MnF_3.

U.S.P. 2,578,913 (2.11.49; 18.12.51). G. M. Whitman, assr to E.I. du Pont de Nemours & Co. Preparation of fluorinated hydrocarbons by heating a mixture of a paraffin, HF, and oxygen at 325° to 700° C in contact with a catalyst consisting of an inorganic salt or oxide of a variable valent metal.

U.S.P. 2,577,388 (22.1.45; 4.12.51). G. W. Warren, assr to Dow Chemical Co. Production of tetrachloroethylene by heating a mixture of an aliphatic compound *A* of formula $C_nHX_{n'}Y_{n''}$ (wherein X and Y = H or Cl, $n = 2$ or 3, n' and $n'' = 2n - 1$ or $2n + 1$), CCl_4 (<0·4 mole of *A*) and sufficient chlorine to perchlorinate *A*, in vapour phase at 500° to 800° C.

U.S.P. 2,579,601 (16.8.50; 25.12.51). C. R. Nelson, M. A. D. Taylor, D. D. Davidson, and L. M. Peters, assrs to Shell Development Co. Production of alcohols by passing a vaporous mixture of an olefin and water through a cat bed comprising H_3PO_4 absorbed on a solid porous support and adding additional quantities of make-up acid to the bed as the process continues.

U.S.P. 2,578,841 (30.1.48; 18.12.51). N. C. Robertson and R. T. Allen, assrs to Celanese Corp. of America. Production of ethylene oxide by passing a mixture of ethylene and air in vol ratio 1 : 10 in contact at 238° C for 1·0 to 1·5 sec with a fluidized catalyst comprising 80-mesh inactive alumina having deposited thereon 43 wt-% Ag, 7·5 wt-% BaO_2, and 0·5 wt-% ZnO.

U.S.P. 2,578,724 (24.11.48; 18.12.51). J. K. Mertzweiller, assr to Standard Oil Development Co. Preparation of high-mol.-wt. unsaturated ethers of branched-chain structure by heating together alkyl C_8 aldehydes and aliphatic alcohols, said ethers consisting largely of C_{16} ether.

U.S.P. 2,579,411 (20.1.50; 18.12.51). R. L. Adelman, assr to E.I. du Pont de Nemours & Co. Production of vinyl ethers by reacting an alcohol with a vinyl ester at $-75°$ to $-15°$ C in presence of a mercuric salt of a strong acid.

U.S.P. 2,579,021 (Neth. 10.7.46; 18.12.51). W. C. B. Smithuysen and C. D. F. Eerbeek, assrs to Shell Development Co. Production of di- and tri- chloro acetaldehydes and their acetals by reacting part of the required amount of chlorine with ethyl alcohol in the cold ($<35°$ C) and then passing the product continuously in countercurrent to remaining amount of chlorine at elevated temp (60° to 100° C).

U.S.P. 2,579,412 (2.3.50; 18.12.51). R. L. Adelman, assr to E.I. du Pont de Nemours & Co. Production of vinyl ethers by reacting at below $-10°$ C in presence of a mercuric salt of a strong acid a vinyl ether with an alcohol or phenol containing from 1 to 3 OH groups.

U.S.P. 2,578,647 (18.10.50; 11.12.51). C. H. Stiteler and P. O. Tawney, assrs to United States Rubber Co. Preparation of esters of 1 : 2 : 4-butanetriol by heating allyl alcohol, or its ester with a saturated aliphatic $C_{>4}$ monocarboxylic acid, with H_2SO_4, HCHO, a saturated aliphatic $C_{>4}$ monocarboxylic acid and a saturated aliphatic C_{3-4} carboxylic acid anhydride.

U.S.P. 2,578,657 (21.8.48; 18.12.51). H. W. Anderson, O. A. Samson, and A. W. Clark, assrs to Shell Development Co. Production of oil-soluble pet sulphonic acids dissolved in a highly refined lub.-oil fraction by sulphonating a lub.-oil raffinate of naphthenic character with fuming H_2SO_4 at 75° to 150° F for *ca* 10 min and, before sludge separates, intimately mixing with *ca* 25 to 80 wt-% water (based on acid) and separating an aq phase from the resulting oil phase.

U.S.P. 2,577,796 (28.11.50; 11.12.51). R. C. Morris, V. W. Buls, and S. A. Ballard, assrs to Shell Development Co. Di-esters of $\beta\gamma$-olefinically unsaturated alcohols of phosphonous or thiophosphonous acids (diallyl benzene phosphonate) and polymers thereof.

U.S.P. 2,576,625 (8.10.47; 27.11.51). R. J. Miller, assr to California Research Corp. Oxidn of durene to pyromellitic anhydride by contacting a gaseous mixture of durene with 50 to 200 times its wt of air with a supported V_2O_5 cat at 850° to 1050° F at a rate such that 0·05 to 3 moles of durene are contacted with 50 to 100 cc of cat per hr.

U.S.P. 2,577,768 (19.7.49; 11.12.51). G. G. Joris, assr to Allied Chemical and Dye Corp. Oxidn of cumene to cumene hydroperoxide in liq phase with O_2 in presence of solid $NaHCO_3$ at 60° to 90° C.

U.S.P. 2,580,300 (12.8.49; 25.12.51). H. L. Johnson and A. P. Stuart, assrs to Sun Oil Co. A polyalkyl benzophenone mixture averaging 19 C atoms per mol and boiling at 392° to 482° F/4 mm is prepared by reacting a pet fraction comprising aromatics and saturates and boiling at 300° to 400° F with phosgene in the presence of $AlCl_3$, and hydrolysing the reaction products. (In U.S.P. 2,580,301 this product is used to plasticize P.V.C.)

U.S.P. 2,578,823 (8.9.48; 18.12.51). V. Molinari and H. G. Affholter, assrs to Union Carbide and Carbon Corp. Preparation of phenol by reacting benzene and oleum in molar ratio at 60° to 80° C in presence of 5 to 10% $PhSO_3Na$ to effect partial conversion to $PhSO_3H$, passing the mixture to a separate zone at 150° to 170° C in countercurrent to benzene vapour until $\leqslant 90\%$ conversion to $PhSO_3H$, neutralizing with Na_2SO_3 at *ca* 105° C, cooling and separating $PhSO_3Na$. This salt is then mixed with molten PhONa and hydrolysed with dry steam to produce Na_2SO_3 and PhOH.

U.S.P. 2,578,597 (22.11.48; 11.12.51). S. P. Robinson, assr to Phillips Petroleum Co. Production of mono-alkyl phenols (*p-tert*-butylphenol) by alkylating a phenol with a low-b.p. olefin in presence of a SiO_2–Al_2O_3 catalyst and an inert diluent, separately recovering mono- and poly-alkyl phenols from the reaction products and reacting the poly-alkyl phenols with phenol in presence of a used SiO_2–Al_2O_3 catalyst at an increased temp to produce additional mono-alkyl phenol.

U.S.P. 2,578,206 (26.9.47; 11.12.51). H. Pines and J. A. Vesely, assrs to Universal Oil Products Co. Production of alkenyl phenols by reacting a phenol in liquid phase with a diene in which one double bond is attached to a tertiary C atom in the presence of a catalyst complex consisting essentially of a lower aliphatic ether and H_3PO_4 at $-10°$ to 100° C.

U.S.P. 2,580,284 (11.4.49; 25.12.51). T. J. Deahl, F. H. Stross, and M. D. Taylor, assrs to Shell Development Co. Production of *sec*-arylamines (*N*-methylaniline) by reacting a $C_{>4}$ alkanol or alkenol (1 to 4 mols) with a primary unsubstituted arylamine (1 mol) at 200° to 350° C/15 to 100 p.s.i. in the presence of a cat consisting essentially of 2 to 20% Cu, an absorptive Al_2O_3 and 0·2 to 4 mols, per mol of Cu, of an oxide of Ca, Mg, Zn, Cr, Mn, or Fe.

U.S.P. 2,578,326 (6.9.49; 11.12.51). W. G. Toland, assr to California Research Corp. Purification of benzene polycarboxylic acids by mixing the solid crude acids with a hydrocarbon oil boiling at 400° to 750° F, heating to below the m.p. of the solid and withdrawing vaporized oil and sublimed acid.

U.S.P. 2,578,654 (28.1.50; 18.12.51). G. W. Hearne, T. W. Evans, and V. W. Buls, assrs to Shell Development Co. Production of *tert*-butylbenzoic acid by oxidizing the corresponding *tert*-alkyl toluenes in liquid phase in the presence of 0·005 to 5 wt-% of a soluble salt of a heavy metal (Co naphthenate) at 130° to 225° C/$\geq$1000 p.s.i.

U.S.P. 2,578,950 (27.5.49; 18.12.51). J. R. Scheibli, R. C. Morris, and E. C. Shokal, assrs to Shell Development Co. Unsaturated esters of *tert*-butyl benzoic acids with C_{2-4} alkenyl alcohols (allyl and vinyl *p*-*tert*-butylbenzoates), their polymers, and compositions. V. P. P.

Coal, Shale, and Peat

1714. Extraction of methane from coal seams. L. T. Minchin. *Times Rev. Ind.*, 1952, **6** (63), 24–5.—Description of procedure at Point of Ayr (Flintshire) colliery. Gas (97 to 99% CH_4) is obtained under pressure (up to 300 p.s.i., controlled to 10 to 20 p.s.i. at manifold) by boring into as yet unworked seams. Daily output has been $\frac{1}{2}$ to 1 million ft^3; at present sole use of gas is for steam-raising at colliery, economizing 240 tons coal/week. Diagram of workings is given. V. B.

1715. Dehydration and improvement of raw peat and raw brown coal. E. Terres. *Brennstoff-Chem.*, 1952, **33** (1–2), 1–12. (*Lecture at Carl-Engler and Hans-Bunte-Institute for mineral oil and coal research, Techn. High School, Karlsruhe, Oct.* 1951.)—Wet peat, brown coal, and similar substances of a colloidal nature are dehydrated economically by a new procedure. *Ca* 100% by wt of H_2O is added to peat, which is comminuted, kneaded to a slurry, and pumped into a reaction vessel. Heating to 200° C under 15 to 20 atm pressure breaks down the colloidal part of the aq-liq phase, and H_2O is separated by filtering or centrifuging. At temp 250° to 270° C exothermic decarboxylation and dehydration of the solid substance occur, forming highly reactive peat charcoal. The heat generated, 400 to 800 k.cal/kg, is used economically by coupling this procedure with the colloid breakdown at 200° C. The peat charcoal may be briquetted, gives a high coke yield and a gas resembling coal gas. High reactivity renders the products suitable for hydrogenation and conversion into power and motor fuels. At temp up to 300° C the peat slurry reacts with H_2O, and an almost N-free mixture of H_2, CO, and CO_2 is obtained. On further heating the hydrogenation reaction sets in, forming a mixed gas of high cal. val. consisting chiefly of H_2, CH_4, and C_2H_6. At 374° C, the crit temp of steam, liq and solid hydrocarbons are formed. The procedure of combining the synthesis of hydrocarbons in the aq–liq phase with the gasification, using H_2 of the gasification gas with or without supplementary H_2 or gases containing H_2, indicates a tech synthesis of gaseous, liq, or solid hydrocarbons in one operation from low-grade fuels. This is of importance in view of the vast peat deposits of the world and the fuel crisis. Extensive tabulations and charts of relevant data are given. R. T.

1716. Scottish shale oil. A review of an important British industry—I. Anon. *Petroleum*, Apr. 1952, **15** (4), 92–5.—Reviews the early history and later developments of the shale-oil industry. The work carried out at the Burngrange pit is described as typical of running operations. The Westwood works is also described as typical of recent shale-oil works, and a brief description of the sulphate of ammonia plant is included. R. E. P.

1717. Shale oil production and refining to-day. Pt I. P. W. Sherwood. *Refiner*, Feb. 1952, **31** (2), 98–101.—The increase in demand for liquid fuels has created new interest in the production of fuels from shale oil. The resources of shale oil are discussed, and it is estimated that excess of 225 million brl of crude oil is obtainable from deposits in the U.S.A., the most important area being in the Rocky Mountains. The classification, properties, and processing of oil shale are discussed. A. R. H.

1718. Shale oil production and refining to-day. Pt II. P. W. Sherwood. *Refiner*, Mar. 1952, **31** (3), 134–8.—Retorting processes for the recovery of shale oil which have been commercially established or could be used on a commercial scale are described, these include fluidized-solid technique, modification of the Thermofor catalytic process, counterflow retorting processes, the Royster retort, and the gas-flow retort. A. R. H.

1719. Hydrogenation of shale oil. W. M. Smith, T. C. Landrum, and G. E. Phillips. *Industr. Engng Chem.*, Mar. 1952, **44** (3), 586–9.—Reported that hydrogenation of a total crude shale oil at 3000 p.s.i.g. over a fixed-bed catalyst gave products which could be treated in conventional petroleum equipment as a synthetic crude oil. Catalyst used was a mixture of nickel and tungsten sulphides. Treatment of distillate fractions was less than required by those from unhydrogenated crude shale oil.

Data on properties of crude shale oil and hydrogenated products are tabulated. The apparatus used was reported in *Industr. Engng Chem.*, 1946, **38**, 136–40. E. J. C.

1720. Composition and conversion of hydrocarbons from low-temperature coal tar. W. Fuchs and K. A. Hamacher. *Erdöl u. Kohle*, 1952, **5**, 80–3.—Composition of tars from low-temp carbonization is discussed. Separation of tar hydrocarbons from other constituents is described. Their classification was effected by a modified Kattwinkel procedure, whereby heat of reaction is diminished by greater dilution with pet ether. Refractometric measurements indicate that neutral oil aromatics are polycyclic. Origin and pyrolysis of low-temp coal tars was investigated. Thermodynamic considerations, confirmed by experiment, show the unstable nature of tar components. Mention is made of work now proceeding on reactivity of coal-tar hydrocarbons. P. V.

1721. Inhibiting properties of tar acids and bases from the oil-shales of Aleksinac on cracked gasoline. J. Sirola. *Nafta* (*Yugoslavia*), Nov. 1951, **2** (11), 3–7.—The acidic and basic components from the tar of the oil shales of Aleksinac (Serbia) have been isolated and tested for their inhibiting action in the auto-oxidation of cracked gasoline, to which purpose precise vacuum-fractionation of the tar has been carried out. It has been found that a particular phenol-fraction greatly prolonged the induction-period of decomposition of cracked gasoline, while pyridine bases exhibited small effect. A suggestion on the use of the phenol-fraction for the stabilization of gasoline stocks is given. (Author's summary.)

Miscellaneous Products

1722. Synthetic detergent production. L. Flett. *Soap Sanitary Chem.*, Mar. 1952, **28** (3), 36.—The U.S. production of synthetic detergents from 275,000,000 lb in 1946 increased to 1,500,000,000 lb in 1951. The two billion mark is estimated to be reached in 1954 with some flattening of the curve. The steep rise in the synthetic detergent production has been accompanied by a rise, now flattening out, in soap production, indicating that the total detergent field is expanding. The availability of raw materials is considered to be more likely to limit expansion than demand. The article also compares the rate of growth of graphs of synthetic detergents with synthetic resins and synthetic fibres. N. W. G.

1723. Soaps and synthetic detergents. J. P. Sisley. *Bull. Ass. franç. Tech. Pétrole*, 1952 (91), 43–52.—French production of syndets in 1951 was approx 50,000 tons, distributed as follows (% active material). Sulphonated fatty alcohols 9000 (30), sulphonated fatty amides 4000 (20), alkylarylsulphonates 10,000 (30), sulphonated secondary alcohols 25,000 (20), non-ionic materials 500 (100), and cationic materials 120 (various concn). Principal advantages of soap over syndet are: greater efficacy in

soft water; easier rinsing; greater dispersant power; stabler foam; fewer difficulties in sewage treatment; less degreasing action on human skin; greater consumer appeal; cheaper price; easier storage. Syndets score on basis of: greater efficacy in neutral or acid media and in hard water; greater variety of types; greater suitability for delicate textiles; greater antiseptic power; greater foaming power; effective at lower concn, and in seawater. Possibility of utilization of soap/syndet mixtures is briefly considered. V. B.

1724. Aerosol insecticides—their evaluation against house flies, cockroaches. A. C. Miller, A. Mallis, and R. V. Sharpless. *Soap Sanitary Chem.*, Mar. 1952, **28** (3), 143.—Aerosols based on synergized pyrethrins were fully tested against house flies, and increasing effectiveness was given by raising the temp (70° to 80° F) and increasing the number of flies in the test group. Dosage increase gives higher mortality for formulæ of poor quality, but if the product is already effective, dosage increase causes little change. The height and manner of discharge and the rate of delivery should be subject to control, though variations in the last item showed no effect on the results. Tests on cockroaches—both the space and direct application—showed a lower mortality for the larger American and Oriental roaches. In general, larger dosages were required than for house flies. N. W. G.

1725. Insect repellents. M. A. Lesser. *Soap Sanitary Chem.*, Mar. 1952, **28** (3), 136.—A review (fifty-seven references) of published formulæ in this field. These products are required to give protection against disease-carrying pests as well as to reduce factors such as annoyance. The ideal properties are that the blend should be cosmetically acceptable, effective for several hours, and should be relatively non-toxic on skin. The older repellents included essential oils such as citronella, but the newer products are odourless, the main ones being dimethyl phthalate, dimethyl carbate, and various proprietary compounds. The formulæ include both liquid and cosmetic paste type of repellents. N. W. G.

1726. Cleaning and maintenance of hardwood floors. F. H. Lyons. *Soap Sanitary Chem.*, Mar. 1952, **28** (3), 44.—In addition to discussing the laying and mechanical surfacing of floors, article discusses some of the materials used in finishing formulations. Of particular interest are the comments on the use of wax-containing preparations. In the initial finish waxing should not be given until the floor is completely dry and, for porous timbers, filling has been applied. Cleaning methods use wax-hosed products to remove stains and re-wax in one operation. In re-polishing only paste and liquid waxes should be used, since self-polishing emulsion types will disturb the moisture content of the wood. As a general principle two thin coats are superior to one thick coat. N. W. G.

1727. Patents. U.S.P. 2,576,515 (18.3.48; 27.11.51). H. L. Johnson and A. P. Stuart, assrs to Sun Oil Co. Polymerization of 4-vinyl*cyclo*hexene to an oily product of visc *ca* 60 cs at 210° F by contacting with a BF_3–ether complex, H_2SO_4, or $SnCl_4$ at 35° to 130° C.

U.S.P. 2,580,184 (26.7.48; 25.12.51). M. J. Murray, assr to Universal Oil Products Co. Drying properties of conjunct hydrocarbon polymers of polyolefinic cyclic structure recovered from the sludge formed in conjunct polymerization of C_3 monoolefins are improved by combining with an equi-molecular proportion of oxygen at −30° to +200° C.

U.S.P. 2,579,007 (29.5.48; 18.12.51). M. J. Murray and W. S. Gallaway, assrs to Universal Oil Products Co. Improving a drying oil comprising a mixture of cyclic, polyolefinic hydrocarbons recovered from a sludge formed in a conjunct polymerization reaction, by contacting with a silica catalyst to effect isomerization–polymerization.

U.S.P. 2,578,214 (31.12.48; 11.12.51). J. P. West, assr to Universal Oil Products Co. Drying oil composition comprising (1) 20 to 95% of a non-drying hydrocarbon oil containing polyolefinic hydrocarbons of mol. wt. <2000 formed by low-temp-catalysed copolymerization of a di-olefin and a mono-*iso*-olefin and (2) 5 to 80% of a mixture of polyolefinic cyclic hydrocarbons recovered from sludge formed in a conjunct polymerization reaction.

U.S.P. 2,578,169 (24.11.48; 11.12.51). H. S. Bloch, assr to Universal Oil Products Co. Copolymers of an ethylenically unsaturated compound with a polyolefinic cyclic hydrocarbon.

U.S.P. 2,580,362 (22.1.49; 25.12.51). D. E. Sargent, assr to General Aniline & Film Corp. Production of thermo-reversible gels by mixing aq polyvinyl alcohol with an aq alkaline solution of 4 : 4′-diaminodiphenyl- or 4 : 4′-diaminostilbene-2 : 2′-disulphonic and dicarboxylic acids in which the amino groups are acylated.

U.S.P. 2,579,219 (18.8.48; 18.12.51). C. J. Vander Valk, assr to Heyden Chemical Corp. A vinyl chloride–vinyl acetate copolymer plasticized with an ester of a pentaerythritol hydroxyalkyl ether in which the acyl and alkyl groups contain 2 to 4 carbon atoms.

U.S.P. 2,578,853 (11.4.50; 18.12.51). J. K. Stevenson, assr to Niagara Alkali Co. Alkoxy chlorobenzenes as plasticizers of butadiene–acrylic nitrile synthetic rubber.

U.S.P. 2,578,688 (25.9.48; 18.12.51). G. L. Fraser, assr to Monsanto Chemical Co. Vinyl-chloride-containing polymers plasticized with trimethylol propane trioctanoate.

U.S.P. 2,577,734 (31.1.51; 11.12.51). H. C. Brinker, assr to Minnesota Mining & Manufacturing Co. Polyvinyl acetate emulsion plasticized with an ester of a polyhydric alcohol and abietic acid having at least one free OH group.

U.S.P. 2,579,375 (4.1.50; 18.12.51). J. B. Eisen, assr to Monsanto Chemical Co. Non-electrostatic polystyrene article containing 0·005 to 5 wt-% of a lipophilic quaternary ammonium salt in which N is attached to hydrocarbon radicals only, $\leqslant 1$ radical containing $\leqslant 6$C atoms and $\leqslant 1$ radical containing $\geqslant 4$C atoms.

U.S.P. 2,577,635 (2.3.49; 4.12.51). E. D. Serdynsky, J. S. Gowing, R. M. Wiley, and C. B. Havens, assrs to Dow Chemical Co. Compositions having little tendency to acquire and retain a charge of static electricity, comprising a vinylidene chloride polymer and 0·5 to 5 wt-% (based on polymer) of a penta-alkyl tripolyphosphate or a hexa-alkyl tetrapolyphosphate, wherein the alkyl groups contain 3 to 8 C atoms.

U.S.P. 2,579,572 (27.6.50; 25.12.51). J. G. Hendricks, assr to National Lead Co. Vinyl chloride resin stabilized to light with Na_3PO_3 or K_3PO_3 and barium ricinoleate.

U.S.P. 2,578,359 (19.8.46; 11.12.51). R. L. Jenkins, assr to Monsanto Chemical Co. Stabilization of halogenated aryl hydrocarbons with dibutyl diphenyl tin.

U.S.P. 2,578,904 (18.3.49; 18.12.51). G. S. Stamatoff, assr to E.I. du Pont de Nemours & Co. Stabilizing chlorinated ethylene polymers by agitating the aq suspension at $\leqslant 50°$ C with minor amounts of propene oxide and a protein.

U.S.P. 2,579,008 (24.5.48; 18.12.51). M. Naps and F. E. Condo, assrs to Shell Development Co. Interpolymer of 65% methacrylonitrile, 25% acrylonitrile, and 10% vinyl acetate.

U.S.P. 2,579,061 (10.7.50; 18.12.51). D. W. Woodward, assr to E.I. du Pont de Nemours & Co. Copolymers of HCN and ethylenically unsaturated monomers in which recurring imino groups are attached to carbon atoms of the polymer chain.

U.S.P. 2,579,079 (12.11.48; 18.12.51). P. O. Tawney, assr to United States Rubber Co. Interpolymers of mono-2-alkenyl ethers of saturated monohydric alcohols or phenols with esters of saturated monohydric alcohols or phenols and α-unsaturated aliphatic carboxylic acids.

U.S.P. 2,579,871 (9.6.49; 25.12.51). D. L. Schoene, assr to United States Rubber Co. Protein of wool is reacted with divinyl sulphone.

U.S.P. 2,576,955 (18.10.46; 4.12.51). N. C. Ludwig, assr to Universal Atlas Cement Co. Low-water-loss cement comprising Portland cement, 0·025 to 5% polyvinyl alcohol *A* (based on cement), and a minor amount of an agent which suppresses the foam-stabilizing characteristic of *A*.

U.S.P. 2,579,380 (5.10.49; 18.12.51). G. O. Funderburk, assr to E.I. du Pont de Nemours & Co. Non-caking detergent powder comprising a synthetic detergent and a minor proportion of an aluminium polyborate.

CORROSION

1728. Corrosion and its control. M. E. Parker. *Oil Gas J.*, 31.3.52, **50** (47), 122.—No. 48 in this series deals with corrosion in mono-ethanolamine solutions used for removal of acid gases from natural and refinery gases. Decomposition and oxidation products of the amine play only a minor role in corrosion.

In glycol-amine systems used for water removal the critical factor is the decreasing alkalinity of the amine with increasing temp. G. A. C.

1729. How six plants cut corrosion rates by 95%. W. C. Uhl. *Petrol. Process.*, Feb. 1952, **2** (2), 190–7.—By the use of a relatively high-mol.-wt. semi-polar inhibitor, corrosion has been decreased to a great extent in a number of refineries and natural-gasoline plants. The inhibitor action is believed to be due to an adsorbed mono-molecular film on the metal surface. It is injected into the stream to be inhibited at around 2 to 5 lb/1000 brl. Data are presented on the degree of protection achieved in six units. These are a debutanizer heat exchanger, overhead vapour lines, condensers on a topping unit, straight-run gasoline lines in a light oils unit, coolers, etc., in a gasoline-plant compression system, condensers and accumulators in a crude flash tower overhead system and trays and walls in a gasoline-recovery plant, and in a steam stripper. Normal protection with ammonia was sometimes continued. D. W. F.

1730. Cathodic protection. K. A. Spencer. *Fluid Handling*, Feb. 1952 (25), 51. (*Symp., Iron & Steel Inst.*, Dec. 1951.)—The mechanism of corrosion of buried structures by concentration cells, electrolytic cells, and galvanic cells is described, with examples drawn from experience in the Middle East.

The sacrificial anode is most useful in low- and medium-resistance soils. Power-supplied systems for high-resistance soils are based on transformer-rectifiers, generators, and ground-bed installations. D. H.

1731. Refinery painting. W. B. Cook. *Refiner*, Mar. 1952, **31** (3), 128–33 (*Nat. Ass. Corrosion Engrs, Corpus Christi, Texas*, Oct. 1951); *Corrosion*, 1952, **8** (3), 93–9.—Problems of surface protection by painting are discussed. Factors to be considered are the nature of the surface, the type of exposure, and the various materials available for protection. A. R. H.

1732. Patents. U.S.P. 2,577,219 (6.7.46; 4.12.51). A. Wachter and N. Stillman, assrs to Shell Development Co. Corrosion of metal surfaces is inhibited by enclosing in a space containing at least two volatile inhibitors of differing volatility, *e.g.*, amine nitrites or nitrosamines.

U.S.P. 2,577,626 (20.6.47; 4.12.51). C. Phillips, assr to Standard Oil Development Co. Corrosion of Cu, Sn, and Zn in contact with a soln containing NH_3, O_2, and H_2O is reduced by adding a petroleum mahogany acid.

U.S.P. 2,578,585–6 (11.8.49; 11.12.51). G. H. Orozco and R. F. Roy, assrs to Gilron Products Co. Composition for application in aq solution to metals forming thereon an adherent, dry film comprises in parts by wt: polyethylene glycol, fatty acid esters of aliphatic alcohols having >4 OH groups or mixtures thereof (*A*) 5 to 30; alkali metal borate 95 to 70. The composition in 2,578,586 comprises a water-sol high-titre soap 5 to 30; *A* 30 to 5, and balance alkali-metal borate.

U.S.P. 2,578,725 (20.10.49; 18.12.51). J. M. Michel and K. F. Hager, assrs to U.S.A. (Secretary of the Army). Corrosion of metals in contact with alcohols, ketones, or aq solutions thereof is inhibited by adding a compound of formula

$$R.SO_2NH.R_1.COOR_2$$

where R = C_{12-20} saturated hydrocarbon radical, R_1 = arylene or alkylene radical and R_2 = H, alkali metal, or ammonium or substituted ammonium.

U.S.P. 2,580,036 (27.11.48; 25.12.51). A. H. Matuszak and J. W. Hand, assrs to Standard Oil Development Co. Rust-inhibiting composition consisting of a mineral lub. oil, 0·01 to 10% of penta-erythritol mono-oleate, and 0·001 to 5% of propylene diamine.

U.S.P. 2,580,288 (28.9.48; 25.12.51). M. Fainman, assr to Standard Oil Co. (Indiana). Anti-corrosion composition consisting of a liquid oleaginous lubricant and 0·001 to 5% of a dialkoxy diaminosilane in which alkoxy groups contain 1 to 20 carbon atoms. V. P. P.

Engines and Automotive Equipment

1733. Fuel injection systems for large engines. P. Jackson. *Motor Ship*, Mar. 1952, **32** (384), 500.—Two types of mechanical fuel-injection systems are in use for large marine engines, the jerk-pump system and the common-rail system. Examples of both systems are described and illustrated, and particulars are given of recent experimental work carried out on the Doxford engine, when consideration was given to the fitting of the Wilson & Kyle compression-pump system. This combined pump and injector has been successfully applied to conversion of air-injection engines, but difficulties were encountered in its application to the Doxford engine. However, it was finally considered that the greatest simplification and benefit would be obtained by designing a compression-operated fuel pump to meet the conditions and in keeping with the existing camshaft and entablature and cylinder construction. This pump incorporates its own fuel metering device, and is arranged for operating a standard C.A.V. Bosch type automatic spring loaded fuel injector. This pump, however, was not so reliable as the common-rail system, and attention was then directed to the possibilities of an air or hydraulically operated pump. Due to air leakage the pneumatically operated pump was not successful, but a hydraulically operated accumulator pump was designed which is provided with a suction valve and a delivery valve which are mechanically operated by a correctly timed eccentric and cam respectively, integral with the eccentric driving the pump ram. U. M.

1734. Large German diesel engines. P. Schuler. *Motor Ship*, Mar. 1952, **32** (384), 504.—Summarizes the post-war development of two-stroke and four-stroke types of engine in Germany, where it is claimed that m.e.p. and piston speed are approx the same as in all other countries. The main features of the development are given under other countries. The main features of the development are given under the following headings: (1) increase in specific cyl output by improving the scavenging system and employing after charging; (2) exhaust-gas supercharging with two-stroke engines; (3) scavenging blower direct-driven from the crankshaft; (4) equipment of the engine for operating on boiler oil; (5) constructional improvements; (6) development of the highly supercharged four-stroke engine. U. M.

1735. Gas turbine of S.T.S. "Auris." Anon. *Fluid Handling*, Feb. 1952 (25), 39–40.—Further details are given of the open-cycle regenerative gas turbine of 1200 b.h.p. to provide 25% of the ship's max propelling power. The set was tested in the shop for performance and reliability on various fuels and analysed for noise. Installation difficulties were due to the space taken by the four main diesel–electric sets and their accessories, one of which was replaced by the gas turbine. The gas turbine proved thoroughly reliable in the 1391 hr of service. D. H.

1736. Worthington type DR engine. V. M. Holmes. *Gas Oil Pwr*, 1952, **47**, 63–6.—These engines have from five to eight cyl of dimensions 13¼ inches bore and 17½ inches stroke, and operate at 450 or 428 r.p.m. The C.R. is 12 : 1, and the b.m.e.p. is 80 (naturally aspirated), 120, or 160 p.s.i. A range of continuous outputs from 525 to 1760 b.h.p. is available, and a 10% overload can be carried for 2 hr. With modifications they are suitable for use with diesel fuel, diesel/gas mixtures, or with high-compression spark-ignition.

Lubrication is by a gear pump in the sump supplying oil to the bearings through drilled passages, and excess oil from the bearings is used to cool the piston.

Details of the engine construction are given.

For operation on diesel/gas mixtures the conventional fuel pump on each cyl is replaced by a dual-plunger pump, in which the pilot plunger injects a fixed amount of oil (5 to 7·5% of the total B.Th.U.s) at all loads. A throttle valve at the air discharge of the turbo-charger is automatically operated by changes in the exhaust temp, thus

ensuring that the air/gas ratio remains constant at all loads. For operation on spark ignition the fuel nozzles in each cyl are replaced by sparking-plugs using 25 to 30 kV, and balanced air-control valves are added to supply the optimum combustion mixture of gas and air at all loads.

Illustrated by four line drawings. H. C. E.

1737. Swiss diesel shunting locomotive. Anon. *Gas Oil Pwr*, 1952, **47**, 56.—The power unit is a Sulzer two-stroke horizontally opposed engine having three cyl of bore 90 mm and stroke 2 × 120 mm and develops 90 b.h.p. at 1500 r.p.m. The power is transmitted through a four-speed gearbox to both axles of the locomotive, which will haul up to 400 tons on the level.

The fuel consumption is 180 grams/b.h.p.hr. H. C. E.

1738. Articulated rail cars for Venezuela. Anon. *Gas Oil Pwr*, 1952, **47**, 67–9.—The power unit is a horizontal direct-injection engine with six cyl of bore and stroke 4·8 and 5·5 inches respectively, giving an output of 100 b.h.p. The C.R. is 15·75 : 1. Each of the two cars is fitted with an engine, both of which are controlled from a position at either end of the set. The control gear is made as a self-contained unit from which connexions to the various units are made by multi-pin plugs. H. C. E.

1739. Gas turbine locomotive No. 18,100. Anon. *Gas Oil Pwr*, 1952, **47**, 57–62.—This unit, rated at 3000 h.p., drives three main generators through single reduction gear and comprises compressor, combustion chamber, and turbine arranged in line. The fifteen-stage axial compressor has a pressure ratio of 5·25 : 1 at 7000 r.p.m. The combustion chamber contains six flame tubes parallel to the machine axis, and each comprises a large jet orifice with a smaller one for idling. Ignition is by high-tension spark, and fuel is supplied at 650 p.s.i. On starting, an electric motor takes the speed up to 2500 r.p.m. and the turbine accelerates to 4000 r.p.m. under its own power. Idling speed is reached after 30 sec, and full power is available after 10 min.

Details of the main generators and of the auxiliary equipment are given. The fuel pump supplies 7·5 gal/min. and the lub.-oil pump 70 g.p.m. at 50 p.s.i. The lub. oil is cooled to 70° F by two air-blast radiators.

The fuel consumption is 0·88 lb/b.h.p.hr, representing an average thermal efficiency of 15·5% of the heat value of the fuel. Of the 3000 h.p., losses and auxiliary equipment leave 2850 h.p. for input to the traction generators, corresponding to 2450 h.p. at the rails. An eighteen-coach train can attain speeds of 85, 41, and 23 m.p.h. on the level and on grades of 1 in 100 and 1 in 50 respectively. H. C. E.

1740. Drilling for oil in Tunisia. Anon. *Gas Oil Pwr*, 1952, **47**, 53.—The Zeramedine No. 1 drilling rig is powered by three 400-b.h.p. Paxman Mark 12RPH engines, which can be arranged to drive either or both of the draw-works or slush pump. Continuous drilling is in operation, and a depth of 12,000 ft is provided for.

Safety devices on the engine ensure that, should the cooling-water temp rise or the lub.-oil pressure fail, the unit is stopped by withdrawing the scoop in the fluid coupling and draining the oil from the governor gear. Butterfly flap valves are fitted in the air-inlet manifolds, so that if natural gas escapes in quantity the engines can be prevented from running without governor control. H. C. E.

1741. Design features of a 250 kW gas-turbine engine. R. R. Peterson and P. G. Carlson. *Mech. Engng*, Mar. 1952, **74** (3), 197.—Description of an open-cycle gas turbine supplied to U.S. Navy by Solar Aircraft Ltd. as an emergency prime-mover. Air mass flow is 6·6 lb/sec, specific fuel consumption is 1·07 lb/h.p/hr, pressure ratio is 4·8, and max gas temp is 1540° F. The ten-stage axial compressor, with peak adiabatic efficiency of 85% and design point efficiency of 80%, has investment-cast stainless-steel rotor and stator blades. A single aircraft-type combustion chamber is arranged alongside and parallel to the compressor, the combustion intensity at full load being approx 5 × 10^6 B.Th.U/hr/cu. ft/atm. One variable-area burner is used, and ignition is provided by a conventional gas-turbine sparking-plug. The turbine has two stages, with air-cooled disks. Starting is accomplished by compressed air, and the set can be run up to full speed in 17 sec. T. T.

1742. Dale generating set. Anon. *Gas Oil Pwr*, 1952, **47**, 69.—The diesel engine of this set has four cyl of bore 3⅛ inches and stroke 4 inches, and develops 19·5 or 26·5 b.h.p. on the 12-hr rating at 1200 or 1500 r.p.m. respectively. It is directly connected to an 18-kVA generator, and the output voltage is controlled within 2½% without the use of any automatic voltage regulator. The set is intended to serve a small works or as a stand-by unit. H. C. E.

1743. Peat-fired closed-cycle gas turbine. Anon. *Gas Oil Pwr*, 1952, **47**, 55.—This experimental turbine develops 500 b.h.p. and has completed 50 hr running on peat. The peat drying and combustion system is separated from the turbine closed circuit, in which air leaves the compressor at 50 p.s.i. and is heated to 675° F in the heat exchanger and to 1200° F in the air-heater. After passage through the turbine the air returns to the heat exchanger and is further cooled to 75° F before entering the compressor. The combustion system of the air-heater is fired by a peat/air mixture in which the peat is previously dried by waste heat from the turbine.

The comparative efficiency of the plant can be assessed by the statement that the output is greater per 2 lb peat solids than with 1 lb oil fuel. It is hoped that a consumption of <2 lb of peat solids per kWh will be achieved. H. C. E.

Safety Precautions

1744. Better excess flow valves for compressed gases. D. D. Buttolph and H. R. Zeigler. *Refiner*, Feb. 1952, **31** (2), 107–10. *Petrol. Engr*, Mar. 1952, **24** (3), C21–4.—The various types of excess-flow valves, to cut off unrestricted flow in pipes in the event of a breakage, are described. The early designs were maintained open by gravity and could be installed only in the vertical position. Modern types are maintained open by spring pressure, and permit much higher flow rates. Factors affecting the selection of excess-flow valves are discussed; the rating should exceed the desired flow by 10 to 30%, and the position, vertical or horizontal, will slightly influence the capacity. Length of liquid-propane lines protected by valves are given for valves in pipes up to 3 inches. A. R. H.

1745. Protection in control and distribution of power for oil well pumping. C. R. Olson. *Petrol. Engr*, Feb. 1952, **24** (2), B62.—Precautions necessary to protect personnel and equipment in electrical-powered systems are described. C. G. W.

1746. Safety in the dissolved acetylene industry. H. Lagarde. *Chim. et Ind.*, 1952, **67**, 303–13.—Review. History of industry since its inception in 1896 is considered, properties of dissolved C_2H_2 and explosibility of C_2H_2 in various forms discussed. Types of porous packings for C_2H_2 cyl and French regulations and safety precautions are described; max permissible pressure in C_2H_2 cyls is 15 kg/cm², of which partial pressure of C_2H_2 is 13 kg/cm² max. Use of solid C_2H_2 and of liquid C_2H_2 incorporating stabilizers, is briefly mentioned, but these forms have not yet reached the safety level of dissolved C_2H_2, with which no accidents have occurred in France during last twenty years. V. B.

Miscellaneous

1747. An economist looks at the oil industry. G. Tugendhat. *Bull. Ass. franç. Tech. Pétrole*, 1952 (92), 17–39.—Oil economics are considered, especially from the European aspect. Past conditions, enabling industry to develop, and recent changes are discussed. Effect of rising product quality on refinery operation is considered and suggestion is made that reversion to simpler products (*e.g.*, topping in place of cat cracking), with considerably increased fuel oil yield, might prove attractive. V. B.

1748. World benzene situation and its bearing on the future development of the chemical industry. D. W. van Krevelen. *Chem. Wkbld*, 5.4.52, **48** (14), 202–8. (*First Plastic Convention, Arnhem*, Jan. 9, 1952.)—World production and consumption figures of benzene are given. Review of technical production methods: coal carbonization, cracking of heavy petroleum fractions (Catarole process), reforming of light petroleum

fractions. Other possible production methods are coal hydrogenation and coal carbonization without production of metallurgical coke. The economy of the processes are discussed. Forecasts on benzene production and consumption. The possibility of the substitution of benzene by other compounds has been mentioned.
M. M. J. J. S.

1749. U.S. production. Anon. *Chem. Engng*, Feb. 1952, **59** (2), 143–90.—A review of the chemical industry in the U.S. includes technological advances in cracking and reforming, use of lignite, separation of aromatics, and production of phenol from cumene. The demand for benzene is expected to double in the next three years, the petroleum industry supplying most of the increase. Supplies and demands for other hydrocarbons, fats, and oils for 1952 are expected to increase steadily. A list of new plants being erected, planned, or completed during 1951 covers atomic, chemical, metal, petroleum, pharmaceutical, and synthetic-fibre industries. D. H.

1750. French production and refining statistics for 1951. Anon. *Rev. Inst. franç. Pétrole*, 1952, **7**, 51–2.—Production totalled (000's metric tons) 376 (France 293, N. Africa 83); natural gas produced was 285 million m³. Refinery throughput in France for year was 18,462 (individual figures for thirteen refineries given). Refining capacity at beginning 1952 was 20,175. V. B.

1751. Role of cost accounting in refinery operating control. E. M. Skinner. *Oil Gas J.*, 7.4.52, **50** (48), 117.—Four aspects are dealt with. Under economic studies, process problems and plant-operation evaluation are dealt with; operator's accounts are concerned in the cost-accounting system; data grouping and types of accounting are studied under accounting for operations control, and the accounting system includes subdivision of accounts, accumulation of expenses, expense statements, prorating costs, production value, input cost, and cost control. G. A. C.

1752. Want to encourage inventors? R. F. Adams. *Petrol. Process.*, Feb. 1952, **7** (2), 175–7.—The objects of the Sinclair Plan, as announced in May 1951 by Sinclair Research Laboratories Inc., are briefly reviewed. It is based on the supposition that the way to make new ideas more available to companies is to make them more valuable to the individual, who alone is capable of conceiving them. To this end the Sinclair company has placed its research facilities at the service of individuals submitting ideas, related to petroleum products only, for testing them. If the test programme proposed by Sinclair is carried out, Sinclair benefits to the extent of receiving a royalty-free right to use the idea within its companies. D. W. F.

1753. Scrap drive with a profit. W. C. Uhl. *Petrol. Process.*, Feb. 1952, **7** (2), 183–5.—The scrap drive inaugurated at the Markus Hook refinery of Sun Oil Co. in June 1951 is described. Obsolete buildings, process units, boiler units, storage facilities, etc., have been demolished. A small profit was realized on the sale of the scrap steel, apart from considerable amounts of equipment and steel structural members salvaged and place clearance achieved. D. W. F.

1754. Standardization of mineral oils and fuels. A. Beyer. *Erdöl u. Kohle*, 1952, **5**, 174–6.—Historical background since first standardization (in Germany of lub. oils in 1917 is sketched. Work of present standardization body, the Technical Committee for Mineral Oil and Fuel Standardization (Fauchausschusses Mineralöl- und Brennstoffnormung, FAM) is described, and standards issued or about to be issued are listed and briefly discussed. Scope of standards includes both product specification and test procedures. V. B.

BOOK REVIEW

Light Hydrocarbon Analysis. Ed. O. W. Burke, C. E. Starr, and F. D. Tuemmler. *New York: Reinhold Publishing Corp.* London: Chapman & Hall, 1951. 120*s*. Pp. xv + 639.

During the second world war, the need arose, in connexion with the vast synthetic-rubber programme in the U.S.A., for the establishment of rigid specifications and test methods for butadiene and other raw materials. This led to the formation of a body, representing the efforts of Government, academic institutions, and industry, which eventually became known as the Committee on Butadiene Specifications and Methods of Analyses. Recommendations by this Committee were passed to the Rubber Reserve Company (later the Office of Rubber Reserve) which had been created by the Reconstruction Finance Corporation. With the conclusion of the war, the restrictions on publication of scientific information were relaxed, and it was felt by the Butadiene Committee that the enormous amount of information, gained as a result of group effort by many participating companies on methods of light hydrocarbon analysis and butadiene analysis in particular, should be made available for general use. The present book, which is a co-operative effort by the Butadiene Committee, is the result.

Chapter 1 summarizes the history and purpose of the Butadiene Committee, while Chapters 2 and 3, which have been included primarily for the benefit of schools and universities, describe the various processes employed for the production of butadiene and the reasons for the specifications and applicable test methods. The various impurities likely to be present in butadiene are listed, and their effects on the polymerization reaction are discussed.

Chapter 4 deals in some detail with the schemes of analysis that may be applied to hydrocarbon-gas mixtures. The various possible constituents that could be present in typical fractions obtained from low-temperature distillation of a complex mixture are enumerated, and the physical, chemical absorption, or combustion methods that could be used to determine the individual constituents in each fraction are summarized. The chapter ends with a brief review of methods based on the use of the infra-red, ultra-violet, and mass spectrometers, and shows how some of the methods described can be used to determine the purity of polymerization-grade butadiene. Hydrocarbon impurities, for example, are largely determined by mass spectrometer, preferably on the residual gas obtained after the butadiene has been removed (and determined) in the Koppers–Hinckley apparatus, while other impurities such as water, dimer, peroxides, sulphur, etc., are determined by specific chemical or physical tests.

Chapter 5 describes methods available for sampling hydrocarbon gases, and refers in particular to the very real difficulties that can be encountered when attempting to obtain a truly representative sample of a mixture of normally gaseous hydrocarbons by collection from the liquid phase. Safety precautions when handling light hydrocarbons are covered by Chapter 6. The scope of this chapter is wide, and embraces not only such subjects as the inflammability limits of normally gaseous hydrocarbons and safe methods of handling, storing, and disposing of such materials, but includes also valuable information on the design and treatment of sample bombs, on the use of Dewar vessels, and on possible hazards that can be incurred when using liquid nitrogen.

Chapter 7, which comprises the greater part of the book, occupies 362 pages and sets out fifty-four test methods in full practical detail. These methods, which range from a simple weathering test to the use of a mass spectrometer, are not restricted to the testing of butadiene, but complete the schemes of analysis dealt with earlier in the book (Chapter 4) by providing a comprehensive collection of procedures applicable to almost any mixture of light hydrocarbons. They are arranged in groups as follows:—

(i) Sampling and handling of gases (two methods).

(ii) Low-temperature distillation analysis (four methods).

(iii) Chemical methods that can be applied to fractions obtained by low-temperature distillation, in order to determine total olefins, *iso*butene, etc. (Eleven methods.)

(iv) Spectrometric procedures (infra-red, ultra-violet and mass spectrometer). These include methods for individual C_4 hydrocarbons, acetylenes, butadiene, methane, carbon monoxide, carbon dioxide, etc. (Twelve methods.)

(v) Tests relating specifically to butadiene and the pentadienes. (Nine methods.)

(vi) Tests for non-hydrocarbon impurities in butadiene and other gas mixtures (nine methods). Procedures are given for the determination of sulphur, hydrogen sulphide, mercaptans, chlorine, water, peroxides, oxygen, etc.

(vii) Miscellaneous methods covering such procedures as vapour density of gases, appearance of butadiene, non-volatile residue in butadiene, ammonia in liquefied gases, etc. (Seven methods.)

Many of the procedures quoted are re-edited versions of methods previously issued by the Office of Rubber Reserve, while others have been contributed by oil companies and similar participating organizations.

The final section of the book describes work carried out to evaluate the accuracy of many of the methods given. 886 samples of twenty-one mixtures (including nineteen of known composition) were circulated to ninety-seven laboratories, and Chapter 8 deals with the preparation and circulation of these samples. Chapter 9 covers the statistical aspects involved in assessing the many thousands of results reported by the various laboratories. The accuracy and precision obtained for typical methods are summarized in twenty tables at the conclusion of Chapter 9, and the actual test results reported for the various mixtures are given, for reference purposes, in 114 tables in a separate Appendix.

It will be seen from the foregoing that this is an extremely comprehensive book which should prove invaluable to any gas analyst, whether he is concerned with the academic aspects of the subject or with the more practical point of view of analysis in an industrial laboratory. A point of criticism arises here, however, in that an analyst wishing to use one or other of the methods quoted will also wish to know the degree of accuracy that he can expect to obtain, and such information is not given in Chapter 7, where the methods are set out in detail. Admittedly, for many methods, there is an almost embarrassing amount of information, on the subject of precision, in the voluminous section containing tabulated data at the end of Chapter 9, but the intending user of a method, after spending ten minutes or so of hopeful delving amongst these tables, may be exasperated to find that precision data for the method in which he is interested are not included, after all. The reviewer would have liked to have seen each method provided with a section giving the " repeatability " and " reproducibility " limits that are now so familiar to users of the IP and ASTM methods of test books, and which have the merit of being readily understood by those who have little knowledge of statistical theory.

Occasional references to the literature are given at the conclusion of each test method, together with recommendations as to suppliers of apparatus, tap greases, etc., and here again it is felt that the value of the book would have been much enhanced if these limited references could have been augmented by a comprehensive bibliography of the subject.

The book is excellently printed and copiously illustrated with line diagrams of the necessary apparatus; with one or two notable exceptions, as for example, the microscopically small Fig 7.46 on page 293, these diagrams are remarkably clear and easy to use as constructional drawings. The photographs, however, are disappointing, and although those which have obviously been supplied by the manufacturers concerned give, in general, a reasonable impression of the shape and size of the apparatus illustrated, others, as for example Figs 7.26 and 7.71, offer little help to the analyst who wishes to duplicate the equipment shown. These, however, are minor comments, and there is no doubt that " Light Hydrocarbon Analysis " will fill a long-felt need in petroleum, petrochemical, and many other industrial laboratories. Methods which previously had to be gleaned from many scattered sources are here set out side by side, with full details of apparatus, reagents, procedure, method of calculation, etc., given in each case, so that the book, which is worth having merely for its chapters on sampling and safety precautions, can be used in every sense as a practical, working manual. J. H. D. H.

ADDITIONS TO THE LIBRARY

A Source Book of Technical Literature on Fractional Distillation. Gulf Research Development Co.

This book consists of nearly 500 pages of technical papers on distillation collected originally by the Gulf Research Development Co. for use in graduate instruction among its employees. It is now made available to universities, colleges, and reference libraries as a contribution to the chemical-engineering profession.

Part I is devoted to the fundamental aspects of distillation, and commences with a reproduction of Lord Rayleigh's paper of 1902. There are thirty-two papers in this part, the sections covering batch distillation, continuous distillation, azeotropic distillation, phase relations, and separation processes.

Part II reproduces the twenty papers on hydrocarbon absorption and fractionation-process design methods, by Wayne C. Edmister, which appeared in *Petroleum Engineer*, May 1947 to March 1949.

British Standards (British Standards Institution, London).

BS 434 : 1952. Bitumen Road Emulsion for Penetration (Grouting and Semi-Grouting) and Surface Dressing. Pp. 28. 4*s*. net, post free.

BS 846 : 1952. Burettes and Bulb Burettes. Pp. 29. 4*s*. net, post free.

BS 1428 : Part A.2 : 1952. Nitrogen Combustion Train (Micro-Dumas) Microchemical Apparatus Group A : Combustion Trains for the Determination of Elements. Pp. 12. 2*s*. net, post free.

BS 1428 : Part A.3 : 1952. Halogens and Sulphur Combustion Train (Pregl) Microchemical Apparatus Group A : Combustion Trains for the Determination of Elements. Pp. 10. 2*s*. net, post free.

BS 1428 : Part D.1 : 1952. Burettes with Pressure-filling Device and Automatic Zero. Microchemical Apparatus Group D : Volumetric Apparatus. Pp. 14. 2*s*. net, post free.

BS 1752 : 1952. Sintered Desk Filters for Laboratory Use. Pp. 11. 2*s*. net, post free.

BS 1796 : 1952. Methods for the use of BS Fine-mesh Test Sieves. Pp. 27. 3*s*. 6*d*. net, post free.

BS 1839 : 1952. London Pattern Pulley Blocks for Fibre Rope. Pp. 15. 2*s*. net, post free.

BS 1842 : 1952. Double-ended Open-jawed Spanners for BS Hexagon Sizes. Pp. 12. 2*s*. net, post free.

THE INSTITUTE OF PETROLEUM

NOTICES, JUNE 1952

ENTRANCE FEES AND SUBSCRIPTIONS

The current Entrance Fees and Annual Subscriptions for membership of the Institute are :

	Entrance Fee	*Annual Subscription*
	£ *s.* *d.*	£ *s.* *d.*
Members and Fellows	2 2 0	4 4 0
Associate Members and Associate Fellows .	1 1 0	3 3 0
Student Members	Nil	1 1 0

APPLICATIONS FOR MEMBERSHIP OR TRANSFER

The following have applied for admission to or transfer of membership in the Institute. In accordance with the By-laws, the proposals will not be considered until the lapse of at least one month after the publication of this *Journal*, during which time any Fellow, Member, Associate Fellow, or Associate Member may communicate by letter to the Secretary, for the confidential information of the Council, any particulars he may possess respecting the qualifications or suitability of the candidate.

The object of this information is to assist the Council in grading the candidate according to the class of membership.

The name of candidates' proposers and seconders are given in parentheses.

Application for Membership

BARK, William Robert, process plant operator, Anglo-Iranian Oil Co. Ltd. (*D. Blair Watt ; G. Sell.*)

BENNETT, John Albert Reginald, student, University of Birmingham. (*F. H. Garner ; F. Morton.*)

BLACKWOOD, Raleigh Keay, construction superintendent, Kuwait Oil Co. Ltd. (*R. M. S. Owen ; E. Boaden.*)

BONES, Dennis Edmund, operator, Anglo-Iranian Oil Co. Ltd. (*A. R. Stark ; D. Blair Watt.*)

CAESAR, Julius, engineer, Vacuum Oil Co. Ltd. (*S. J. M. Auld ; H. M. Davies.*)

COLEMAN, Michael Coleman, student, University College, London. (*M. B. Donald ; M. Calderbank.*)

CONNOLLY, Ivan Derek Charles, exploitation engineer, United British Oilfields of Trinidad Ltd. (*R. P. Brown ; R. Mackilligin.*)

CRAYSTON, George William, laboratory analyst, "Shell" Refinery and Marketing Co. Ltd. (*A. Crossfield ; H. E. F. Pracy.*)

DAVEY, Peter Patrick, exploitation engineer, United British Oilfields of Trinidad Ltd. (*J. L. Andrews ; R. Mackilligin.*)

EDLER, Dr. Ing. Emil, chief of laboratory, Swedish Gulf Oil Co. (*L. Ubbelohde ; G. R. Schultze.*)

ESCOTT, William Alfred Thomas, general manager and secretary, Messrs. Carless, Capel & Leonard Ltd. (*A. J. Goodfellow ; J. M. Leonard.*)

FROST, Victor Henry, labour superintendent, Apex (Trinidad) Oilfields Ltd. (*H. Gilmour ; H. W. Reid.*)

HARGREAVES, Eric, physical chemist, Trinidad Leaseholds Ltd. (*J. M. Tait ; A. S. Curlet.*)

HARROP, George Newton, drilling engineer, Trinidad Leaseholds Ltd. (*P. E. T. O'Connor ; H. C. Devenish.*)

HAY, Keith Baird Gatacre, plant shift operator, United Coke & Chemicals Ltd. (*D. Blair Watt ; B. D. Cauthery.*)

HENRY, Kenneth George, deputy superintendent (Control Instrument Services, Oilfields), Anglo-Iranian Oil Co. Ltd. (*P. T. Cox ; J. L. Jackson.*)

HERRERA, Donald Anthony, production foreman, United British Oilfields of Trinidad Ltd. (*R. Mackilligin ; R. P. Brown.*)

KEARNEY, Nicholas Anthony, area engineer, Shell Co. of Singapore Ltd. (*E. J. Sturgess ; D. R. Howgill.*)

KING, Philip James, student, Birmingham University. (*F. H. Garner ; F. Morton.*)

KITCHIN, Peter John, lecturer, R.A.F. Technical College. (*R. E. Griffiths ; P. F. Ellis.*)

KOELMAN, Adrianus Dirk, commercial manager (Chemical Industry Management), Shell Petroleum Co. Ltd. (*M. A. Matthews ; R. G. Aickin.*)

KNOTT, Charles, Chief Petroleum Officer & Chief Inspector of Weights & Measures, Borough of Poole Corporation. (*G. P. Lloyd ; G. Sell.*)

LITTLEWOOD, Arthur James, job engineer, The Lummus Co. Ltd. (*Tully Shelley ; J. E. Jones.*)

MARTIN, Cyril Stanley, general manager (White Products), Shell-Mex & B.P. Ltd. (*H. W. Hastings ; T. C. Bailey.*)

MITCHELL, William Franklin, general manager (Chemical Industry Management), Shell Petroleum Co. Ltd. (*R. G. Aickin ; H. N. Short.*)

REYNOLDS, Edward Ainslie, tank design engineer, Shell Petroleum Co. Ltd. (*E. J. Sturgess ; D. R. Howgill.*)

RISSON, Arnold Enfield, operating superintendent, "Shell" Refining and Marketing Co. Ltd. (*J. Grant ; J. L. Black.*)

RUSH, Edward Antissell Evans, divisional engineer, Esso Petroleum Co. Ltd. (*R. S. Coulson ; T. G. Kidd.*)

SMEDLEY, Leonard John, engineer, Anglo-Iranian Oil Co. Ltd. (*P. T. Cox ; J. A. Jackson.*)

TERRINGTON, Allen Edward, supervisor, (Plant & Lub. Service Section Industrial Oils), Messrs. C. C. Wakefield & Co. Ltd. (*G. H. Thornley ; S. E. Holmes.*)

TRAYLEN, George James, district supervisor, Shell Caribbean Petroleum Co. Ltd. (*A. Britton ; W. M. MacKay.*)

VERGHESE, Jacob, technical assistant, Burmah-Shell Oil Co. Ltd. (*M. P. McCarthy ; G. D. Ailawadi.*)

WATSON, Dennys, Lieut., O/C bulk stocks, R.A.S.C. (*A. Souter ; P. F. Ellis.*)

WHOLEY, Max Wingfield, Head of Technical Department, Burmah-Shell Oil Co. Ltd. (*M. P. McCarthy ; G. D. Ailawadi.*)

WILLIAMS, Leslie Harry, Shell Petroleum Co. Ltd. (*M. A. Matthews ; R. G. Aickin.*)

WILSON, Kenneth, process engineer, Bahrein Petroleum Co. Ltd. (*W. T. Barrett ; P. Simpkins.*)

Transfers

BEXON, Roger, geologist, Trinidad Petroleum Development Co. Ltd. (*R. Mackilligin ; L. A. Bushe.*) (*Student to Associate Fellow.*)

GERSHON, John Charles, sales director, Ragosine Oil Co. Ltd. (*G. J. C. Vineall ; A. E. Hope.*) (*Associate Member to Member.*)

SHEASBY, Gerald Roy, chemical engineer, Messrs. Foster Wheeler Ltd. (*F. Morton ; E. D. Hibbert.*) (*Student to Associate Fellow.*)

SOLE, Ernest John, engineer (Gas Section), Kuwait Oil Co. Ltd. (*E. C. Masterson ; H. C. Lack.*) (*Member to Fellow.*)

TAIT, Edward James Horrow, divisional engineer, Trinidad Leaseholds Ltd. (*B. G. Banks ; J. A. Hulme.*) (*Member to Fellow.*)

NEW MEMBERS

The following elections have been made by the Council in accordance with the By-laws.

Elections are subject to confirmation in accordance with the By-laws.

Transferred from Members to Fellows

HOWGILL, D. R. MORLAND, J. PEPPER, A. C.

Transferred from Associate Fellow To Fellow

DUEGAN, B. S.

As Members

CULLINAN, G. H. GODFREY, E. G.

Transferred from Associate Members to Members

ANDREWS, D. CUNNAH, E. MACDONALD, R.

As Associate Fellows

BOULTON, H	DANIELS, W. L. C.	O'DONNELL, G. J.
BRADLEY-KIDD, M.	DUNN, G.	OGDEN, L. F. S.
CARR, S. J.	GILES, D. G.	RUDLING, T.
CLARKE, J. A. R.	GILLETT, H. W.	STIFF, W. J.
COWING, G.	GRANT, W. J.	WIGGINS, R. G.

Transferred from Associate Member to Associate Fellow

SHAWYER, A. C.

Transferred from Student to Associate Fellow

GREEN, R. C.

As Associate Members

GERMAN, W. R. MULHOLLAND, B. D.
MILNER, R. PRICE, W. H.

As Students

BAGGRIDGE, M. T.	GEEN, E. D.	NORMAN, J.
COX, A. G.	GRIFFITHS, R.	PAYNE, F. G.
CUNLIFFE, B. W. P.	HOLLAND, R. A.	SHEARN, D. B.
FRANCIS, D. N.	MAINWARING, A. H.	

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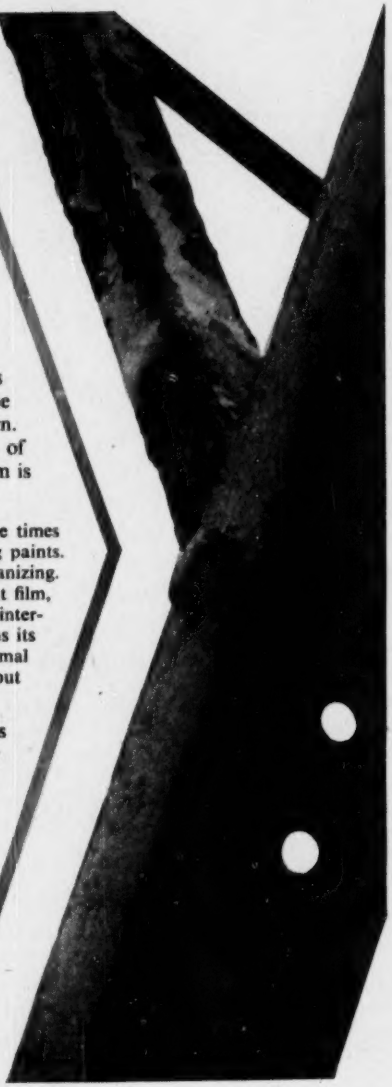

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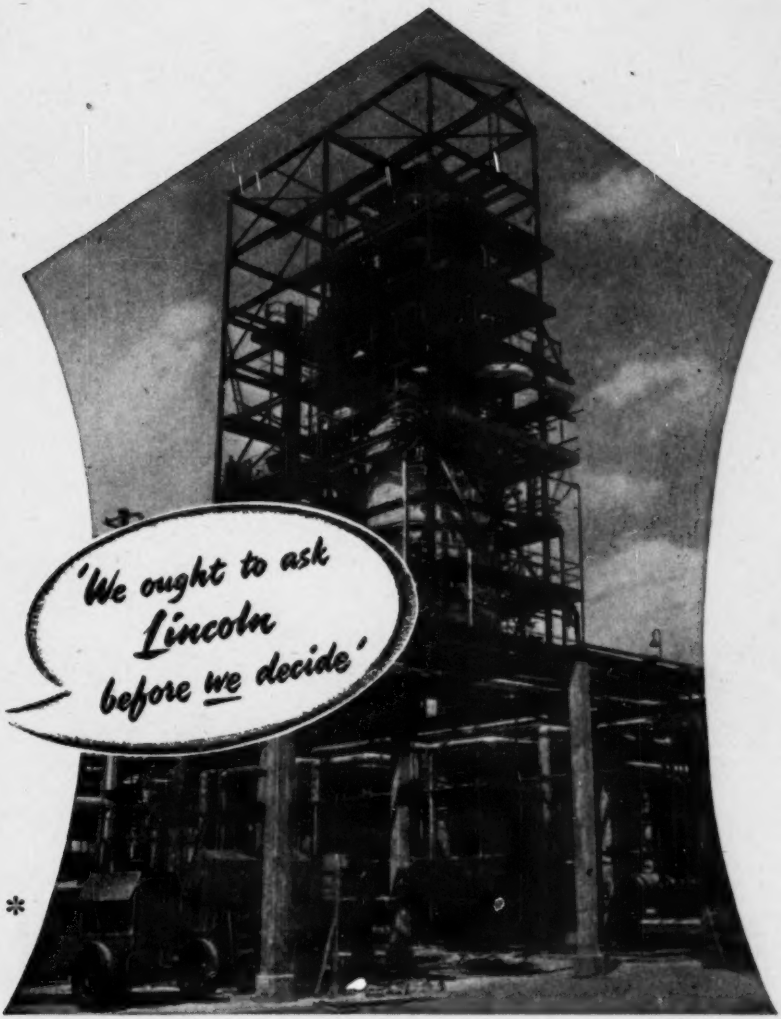

A Shell photograph

* *A Lincoln 300 Diesel—one of the Lincoln machines which with Lincoln electrodes have been used in the construction of this Feed Preparation Plant in the Shell Refinery at Shell Haven*

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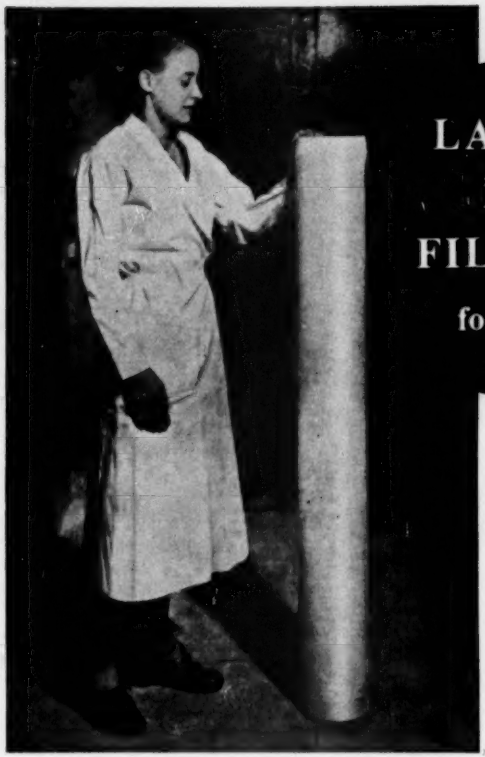

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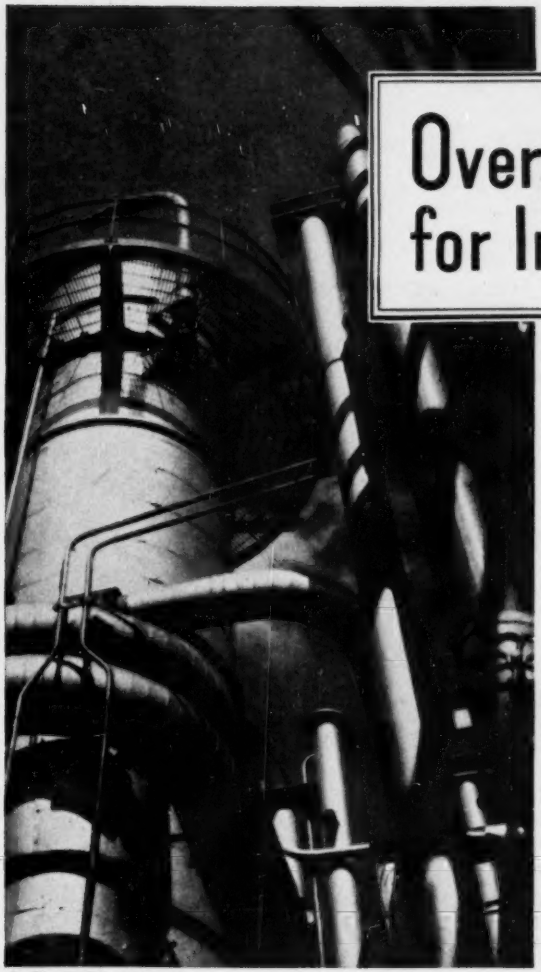
FILTERS



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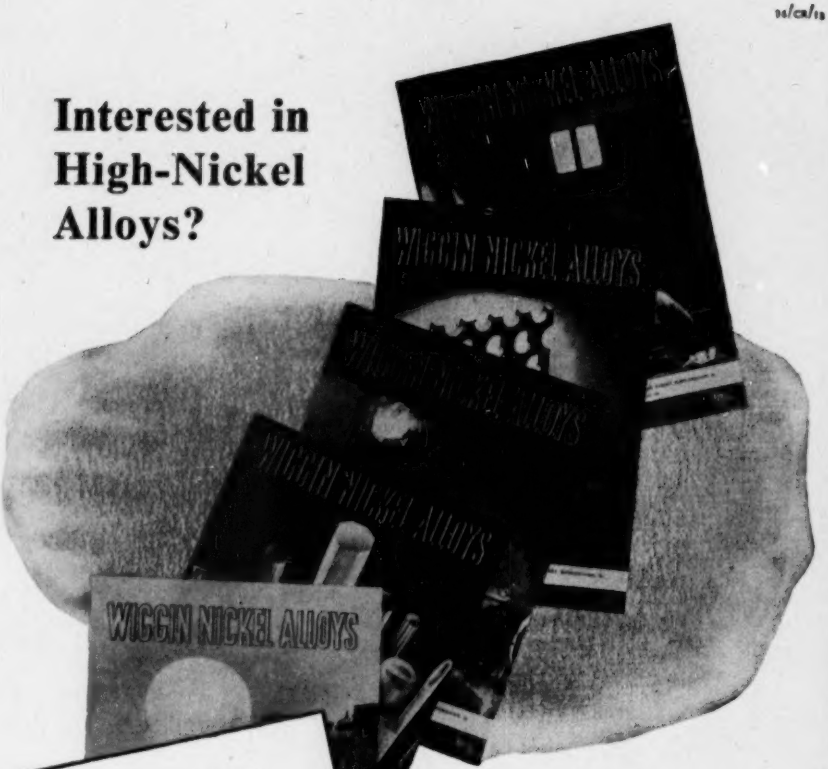

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Monel and Inconel in Fat-Splitting
Monel in Production of Wines and Brines
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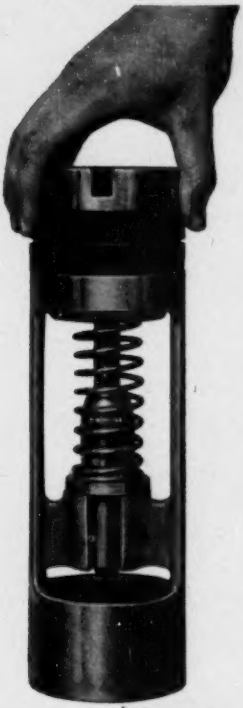

With a BAKER Bakwik Drill Pipe Float

(Product No. 480)

because it brings you these important results:

PREVENTS PLUGGING THE BIT with the hazard and grief of wet strings

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MINIMIZES OR PREVENTS SERIOUS DAMAGE in event of a parted string

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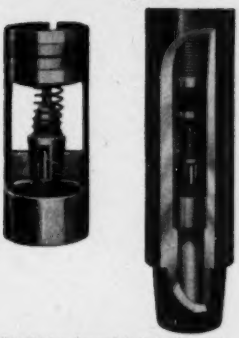

The Valve Assembly is dropped in place by hand and is held there by the tool joint pin above it.

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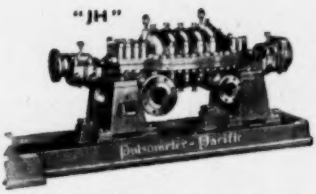
PUMPS FOR THE OIL INDUSTRY

"HV"

Vertically Split Casing Single-Stage Hot Oil Pumps.

"JH"



Horizontally Split Casing Multi-Stage Pumps for Hot Oil.

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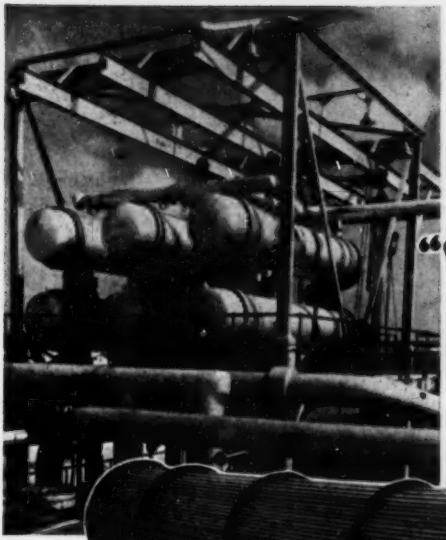

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TUBES
FOR
HEAT
EXCHANGERS
AND
CONDENSERS

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LIMITED
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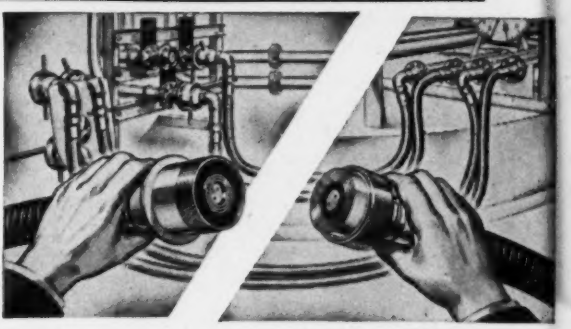
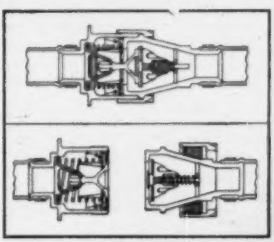
EC1

BREAK . . .
without
spilling . . .



Exactor couplings allow pipe lines to be connected and disconnected as easily as plugging into an electrical circuit—and without the slightest spilling. No air can enter the pipe line. The complication of separate cocks, operated in special sequence, is eliminated. Exactor couplings are automatic, foolproof and suitable for liquids or gases, hot or cold. Write for details.



There is an automatic valve in each half of an Exactor Coupling. When the coupling is joined, the valves mate and keep open by mutual pressure. When you uncouple, each valve seals its side of the break. Full range of sizes from ⅛" to 7" bore, for high and low pressures.

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Seal as they break

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FOR THE OIL INDUSTRY

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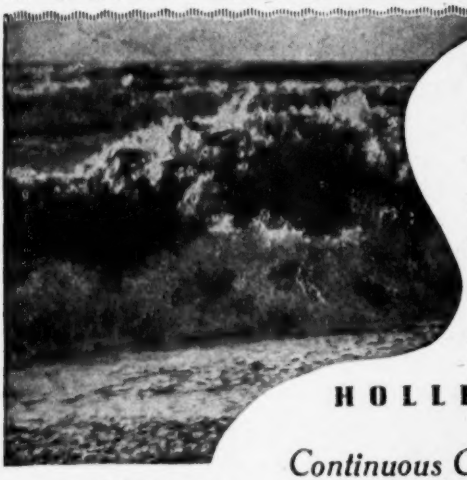

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BRAINTREE ENGLAND

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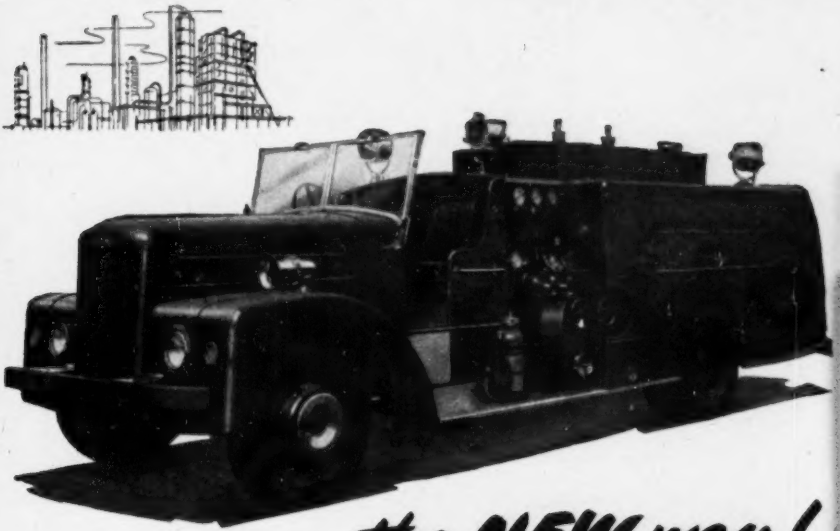

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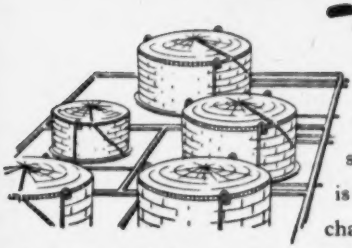

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Pyrene TRADE MARK FIRE APPLIANCES

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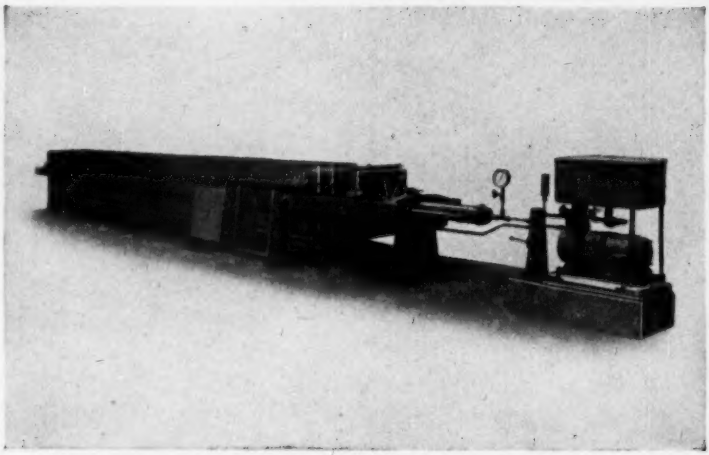

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OUR wide and long experience in the manufacture of wax moulding presses makes us specially suited to meet the most varied demands. May we submit proposals to suit your requirements?

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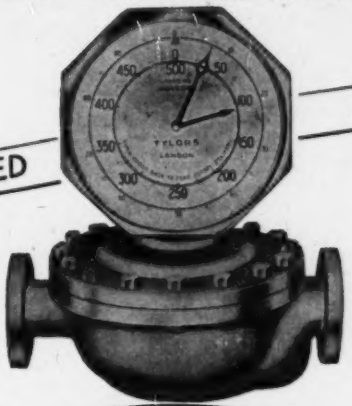

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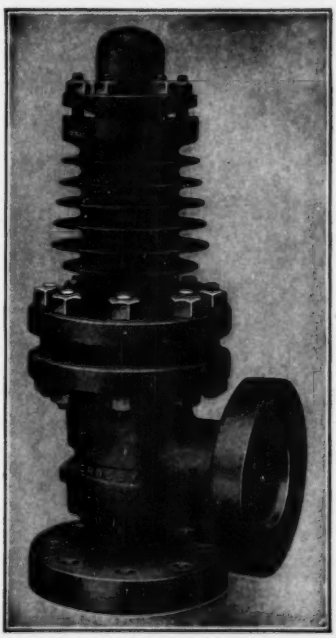

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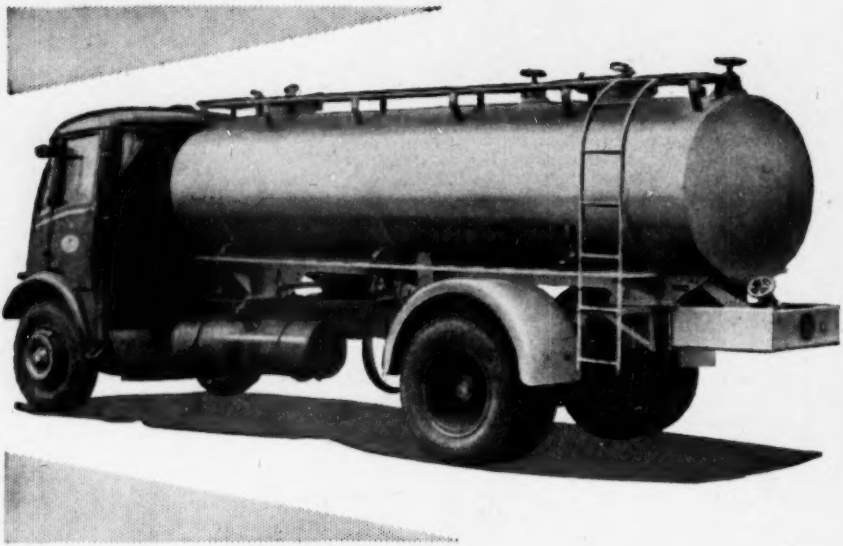

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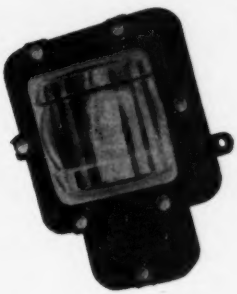
(BUXTON CERTIFIED)



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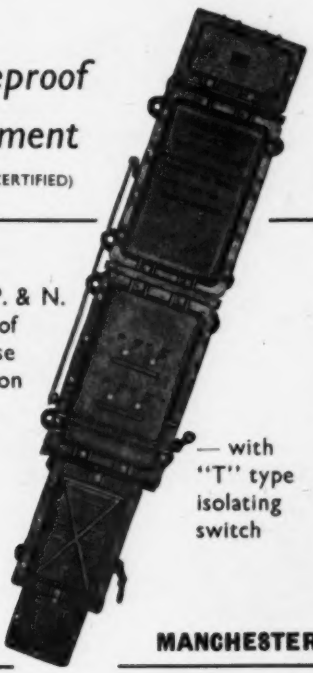
4–way S.P. & N. Flameproof switch fuse distribution board —



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JET NOZZLES
AVAILABLE OVER
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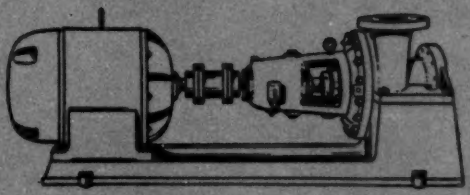
Wilson-Snyder PUMPS for Refinery Service

CENTRIFUGALS -

Type ES Single-Stage Process Pumps



These single-suction pumps are designed with vertically-split case for services where (1) continuous operation is important, (2) temperatures or pressures may be very high, (3) hot or cold liquids are handled near their boiling points and (4) broad capacity range at high efficiency is desirable.
Their design permits maximum interchangeability of parts. Only 3 sizes of bearing brackets, sleeves, packings, shafts and bearings are required for 18 pump sizes; which provide capacity ranges from 40 to 1500 gpm, differential heads from 40 to 1000 feet, speed ranges from 1750 to 4000 rpm, horsepower ranges from 2 to 250, with permissable temperatures up to 900°F.

OTHER TYPES OF CENTRIFUGAL PUMPS . . . include single and multi-stage construction with single or double suctions; providing capacity ranges from 100 to 30,000 gpm and differential pressures up to 1350 psi.

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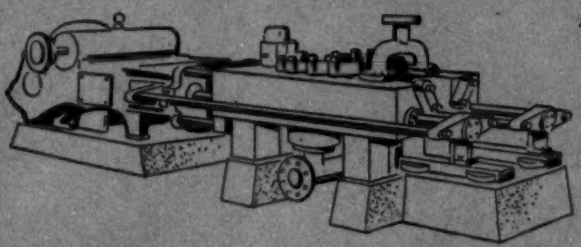

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